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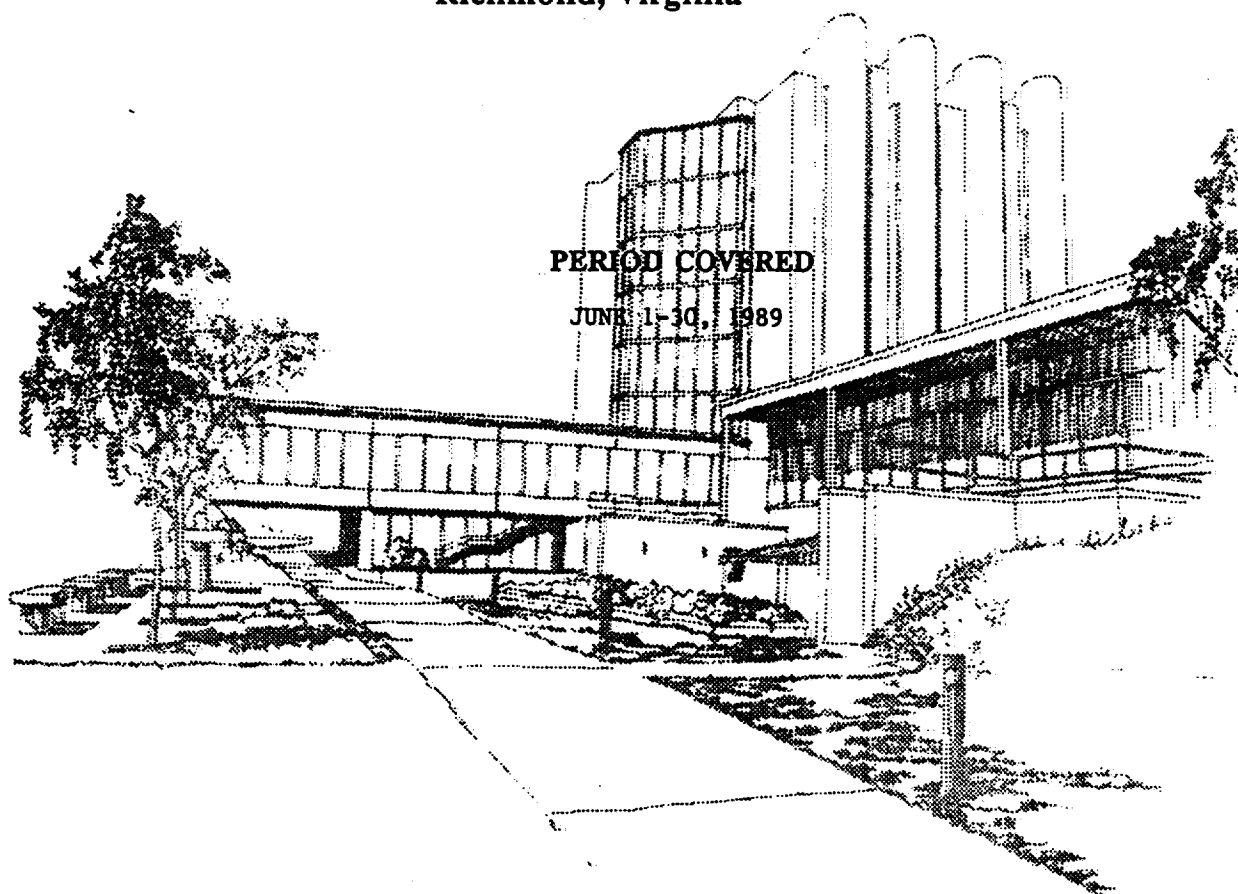
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JUNE 1-30, 1989

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*N/R = No Report

PROJECT NUMBER: 2107
PROJECT TITLE: Filter Research & Development
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: June, 1989

I. IMPROVED FILTRATION - INCREASED EFFICIENCY:

- A. **Objective:** Develop filter systems with a higher efficiency than presently available and evaluate them for subjective advantages.
- B. **Status:** To understand the effect of CA Web variability on subjective response, cigarettes made with a CA Web/CA dual filter were selected according to RTD range and subjectively evaluated. The three RTD ranges of 125-144, 145-160, and 161-180mm of water had FTC tar deliveries of 6.7, 6.1, and 5.7mg respectively. The three models were found to be significantly different subjectively from one another.

Project 605 concentric filter models with 50% filter ventilation were made and achieved acceptable FTC tar deliveries. Samples will be submitted for internal panel testing if Flavor Panel screening is favorable.

Filters were made in the Semiworks with a 1.6/35000 tow item to develop the capability curve. Filters made at the Lark Super Lights RTD target with this tow had a marginal firmness (89%) but performed well during combining.

II. IMPROVED FILTRATION - MENTHOL STABILITY:

- A. **Objective:** Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.
- B. **Status:** Ageing and internal panel evaluation of eleven cigarette models, some with heat treated filters, is complete. Data is now being evaluated and a report is being prepared.

III. IMPROVED FILTRATION - NOVEL FILTER SYSTEMS:

- A. **Objective:** Develop and evaluate new and unique cigarette filters which may offer a distinct product advantage.
- B. **Status:** CONCENTRIC FILTERS: Models of a 3mg Japanese prototype with a 21mm dual charcoal/concentric filter were found to be too high in delivery. Models with a 27mm dual filter will be made and tested.

CA PLUGWRAP: The results of our evaluation of Dexter's CA plugwrap were reviewed with the supplier. The tow-to-plugwrap bonding was unacceptable with the sample we tested. Dexter indicated that they could utilize a different binder system in the

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sheet which may improve bonding to tow. Any further testing is contingent upon receipt of another sample from Dexter.

HERCULES POLYPROPYLENE FILTER TOW: Sample filters made with 3.9/40000 and 5.0/39000 tow items were tested for efficiency, firmness, and other filter characteristics. The filters were low in firmness (86%) but similar to CA filters in other properties. A larger quantity of filters is expected shortly to produce machine-made cigarettes for evaluation.

IV. IMPROVED FILTRATION - SELECTIVE FILTRATION:

- A. Objective: Explore the use of specific additives in filters for selective filtration or subjective modification of smoke.
- B. Status: Cigarette models with FML filters containing a new group of additives for subjective modification continue to be evaluated.

Filters containing a mixture of ethyl citrate esters which are compatible with triacetin are being made this month for analytical and subjective evaluation on GCC Marlboro and Lark cigarettes. Earlier studies with this additive had shown a 15% reduction in nicotine-to-tar ratio.

Several samples of carbon treated with acidic and basic additives were prepared for use in carbon/CA dual filters. These filters will be tested on 8mg ART models.

Carbon samples from three suppliers -- Calgon, Sutcliffe Speakman, and CECA -- are being tested as alternatives for single sourced materials. Cigarette samples have been made with the experimental carbons and their respective controls; analytical smoking, is being conducted.

V. FILTER SUPPORT FOR OTHER R&D PROGRAMS:

- A. Objective: Provide design assistance and potential new filter systems for other R&D programs.
- B. Status: CARBOWAX REPLACEMENT: Danchi test results of four models of PM Super Lights indicate that there was no significant difference in liking scores for the triacetin vs. Carbowax models. Models are now being made for a second Danchi test with slight changes to triacetin application levels.

Four new models of Parliament Lights 100's consisting of variations of Carbowax, triacetin, black tow, and white tow were approved and shipped for Danchi testing.

The Japan Product Development Group is now coordinating the fabrication of Merit Lights and L&M Milds models with triacetin for Danchi testing.

Carbon-on-paper filters from Baumgartner in a PM Super Lights configuration are being tested after two cycles of accelerated ageing. FTC tar deliveries with the paper filters are 5-6mg with 35% filter ventilation. Gas phase results after ageing are not complete.

ALTERNATIVE PLASTICIZERS: Six potential plasticizers were approved for investigation and have been ordered. Only one, diethyl malonate, has been received; testing will begin when all six are available.

LARK VENTILATION VARIABILITY STUDY: Sample bobbins of heat sealable porous combining wraps were received from Dexter and Kimberly-Clark and are being evaluated for reducing the variability of filter ventilation on Lark Milds.

Models of GCC Lark, Lark Milds, and Lark Super Lights were made with Hauni laser ventilation and tested for ventilation variability. The coefficient of variation of filter ventilation ranged from 6 to 11% for the Hauni laser models compared to 24% for standard production Lark Milds KS. Hauni laser perforations are more obvious on the cork-tipped cigarette than the current microlaser perforations.

PROJECT NUMBER: 2108
PROJECT TITLE: New Product Technology
PROJECT LEADER: W. T. Callaham
PERIOD COVERED: June, 1989

I. SAUNA

- A. Objective: Develop products utilizing a modified plastic fluted filter.
- B. Status: An off-line prototype device for blocking the flutes of the plastic filter was fabricated and samples were made. This device melts a groove around the filter and then builds up the melted plastic into the flutes. Conventional Sauna cigarettes (no charcoal section) were made using these filters and were smoked at ISO conditions. The deliveries were the same as for the model with flutes blocked via hot melt bead.

One thousand of the most current Sauna cigarettes (hot melt bead blockage) were delivered to EEMA for testing.

- C. Plans: Sauna cigarettes will be made again via hot-wire melting and hot melt bead to confirm the desired effect. If successful again, PM Engineering will be requested to provide scale-up of this approach.

II. HUMIDOR

- A. Objective: Develop a moisture release device for use in a cigarette pack which maintains pack OV at the desired level.
- B. Status: The final results of the consumer test in U.A.E. have not been received, yet.

In the meantime, at the request of EEMA Marketing, a shipping/ageing study of humidor cigarettes in the Middle East is being designed.

- C. Plans: Conduct the Middle East shipping/ageing study.

III. AMBRASIA

- A. Objective: Evaluate the use of flavor-release compounds in cigarette paper and sideseam adhesives.
- B. Status: Visually acceptable Trim-V cigarettes were prepared using the outer paper coated with Aromatek-245 solutions (3.7% and 1.85% in ethanol). Cigarettes were made with the coating on both the inside and outside of the wrapper. Some of the samples were given to Analytical Research for development of an analytical procedure for Aromatek.

Standard Marlboro lap seam adhesives were supplied by National Starch incorporating ethyl vanillin and Aromatek-245 at a 3.5% level. Marlboro and Va. Slims cigarettes were made with these adhesives and submitted for testing. The Marlboro cigarettes were made at a machine speed of 4200 cigarettes/minute (maximum possible) without any lap seam failures.

- C. **Plans:** Evaluate current models and new cigarettes made with flavored adhesives provided by National Starch.

IV. KAYMICH APPLICATOR

- A. **Objective:** Evaluate products mentholated via the Kaymich application system.
- B. **Status:** A study was completed comparing MF Lights 100 cigarettes using foils mentholated via the Kaymich applicator (molten menthol) and the PM mentholator (menthol/ethanol solution). No significant differences were observed in the smoke deliveries or subjective responses between the test and control cigarettes. However, a spotting analysis conducted after 22 days of lab storage indicated 28 spots/2000 control cigarettes and 52 spots/2000 test cigarettes. Provided spotting is not a problem, molten menthol application to foil appears to be a viable approach for mentholating cigarettes.

Another study was initiated comparing cigarettes (filler) mentholated via Kaymich at the maker versus spray-applied and menthol-on-foil controls. Analytical, subjective and cigarette spot testing is being conducted weekly for 30 days.

V. ASH TRAY ODOR

- A. **Objective:** Develop products which facilitate the elimination of ash tray odor.
- B. **Status:** Spent filters from machine smoked Ambrosia cigarettes (with Aromatek-245) were submitted for testing by the Odor Panel.
- C. **Plans:** Design and conduct an experiment to determine the effect of Aromatek-245 (and other flavors) on ash tray odor.

VI. MACHINERY DEVELOPMENT

- A. **Objective:** Develop or modify equipment to facilitate the development of new products.
- B. **Status:** A PZ application booth was acquired and is being modified to apply liquid additives to CA on the Mark-6/Mini plugmaker.

A CA web/paper plugmaker was placed in the Filter Development Lab and is currently being set-up to run.

A new paper slitter was set-up on the laboratory embossing unit and preliminary samples were prepared. Additional modifications are required to bring the unit up to full capability.

- C. Plans: Complete current equipment modifications.

PROJECT NUMBER: 2304
PROJECT TITLE: Flavor Development/Domestic Product Development/Technology Support
PROJECT LEADER: G. N. Yatrakis
PERIOD COVERED: June, 1989

I. MENTHOL RELEASE COMPOUNDS:

- A. Objective: Develop a mentholated charcoal filtered cigarette utilizing a menthol release compound. Also, to apply menthol release technology to other areas.
- B. Status: Prototypes have been made using the Natural blend C and PMSL blend with MGC to achieve a 0.20% menthol in smoke. Analytical and subjective evaluation is in progress.
- C. Plans: Develop compatible aftercuts and make samples for subjective testing.

See New Product Development, 2304, 4015 for cooperative projects.

PROJECT NUMBER: 2305
PROJECT TITLE: Applied Flavor Investigation
PROJECT LEADER: J. Swain
PERIOD COVERED: June, 1989

I. PROJECT ART

ART-By-Product Utilization:

- A. Objective: To evaluate and develop process modifications for the utilization of ART process by-product tobaccos.
- B. Status: After subjective evaluations of DIET containing 5% ART filler ripper-shorts showed no obvious differences, utilization was recommended for up to 2.5% ripper level in DIET. A ripper test was run at MC to determine potential nicotine transfer to ART filler ripper-shorts. Samples were submitted for analyses.

Utilization of up to 5% OTM (Class A) from Bermuda Hundred in RCB has been recommended based upon results of RCB trials. Cigarettes are being made to select RL and RCB combinations incorporating ART stem from Bermuda Hundred for Stockton Street trials.

C. Plans:

Complete Pilot RL evaluations prior to Park 500 trials	July, 1989
POL test ART stem in RL's and RCB	August, 1989

II. OPERATIONS SUPPORT

PROJECT GRAIN:

- A. Objective: To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.
- B. Status: Analyses of sonolated Cambridge aftercut made at 20th Street with 30% alcohol reduction were within acceptable limits. Semiworks trials of control and test aftercuts have been completed. Cambridge cigarettes submitted for analytical have shown comparable results. Subjective tests are in progress.

An emulsified Marlboro-type aftercut has been submitted for small-scale application in Semiworks trials to evaluate further alcohol reduction.

C. Plans:

Pol Cambridge tests from Stockton Street	August, 1989
Marlboro Evaluation with Emulsion Agent	July, 1989

DRY FLAVOR REPLACEMENT

- A. Objective: To develop, evaluate and establish specifications for dry flavor replacements.
- B. Status: Analyses of larger scale samples from three vendors of RSJB showed two to be comparable in sugar levels. Lab-scale evaluations also showed the two samples to be closer to the control in subjective attributes. Further tests in progress to establish specifications. Pilot RLB trials being planned prior to Park 500 tests.
- C. Plans:
- | | |
|-------------------------|------------|
| Pilot RLB Tests | July, 1989 |
| Finalize Specifications | July, 1989 |

PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization and International Support
PROJECT LEADER: W. R. Bell
PERIOD COVERED: June, 1989

I. MARLBORO STANDARDIZATION

- A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status: Compounding of seven preblends for the Marlboro flavor system was observed. GC analyses are complete for five of the preblends and the raw materials used in compounding. Sample procedures for Standard Run VII were finalized and a report was issued. All of the supplies needed for the factories have been assembled. All analytical and subjective data for Marlboro Lights April 17-18 pick-up have been completed and a report will be issued. Subjective and analytical evaluations are underway on June 13 Marlboro pick-up. Plans for Standard VII Run are complete.
- C. Plans: Marlboro Standard Run VII scheduled for week of July 17.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. Objective: To provide subjective direction for programs with R&D and manufacturing locations.
- B. Status: Fifteen panels completed for the reporting period. Subjective evaluations of factory production start-up for Marlboro Ultra Lights are ongoing.
- C. Plans: Provide assistance as needed.

III. INTERNATIONAL SUPPORT

International Brands Smoking Panel

- A. Objective: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. Status: Thirteen panels completed during the reporting period as well as three brand profiles and evaluations of Marlboro/Marlboro Lights for Japan.

LARK DELUXE MILDS MENTHOL (JAPAN)

- A. Objective: To produce a menthol version of Lark Deluxe Milds to compete with YSL menthol in the Japanese market.
- B. Status: Analytical and subjective results are complete.

- C. Plans: Investigate possible use of mentholated foil for future prototypes.

PROJECT RE-ENTER (CANADA)

- A. Objective: Development of a blend, casing and aftercut system to be used for all Canadian Export Brands.
- B. Status:
- 1) Flavor transmittal made on May 30, 1989.
 - 2) Casing and aftercut produced at the Flavor Center.
 - 3) Filler produced at Stockton Street on June 22, 1989.
 - 4) Cigarette production commencing at all locations, starting at Stockton Street on June 23, 1989.
- C. Plans:
- 1) Analyticals on cigarettes.
 - 2) Subjective evaluations of cigarettes.
 - 3) Commence regular production of Canadian exports as soon as analyticals and subjectives will permit.

PROJECT RING (KOREA) - BRONZE

- A. Objective: Development of a product to be competitive with Eighty-Eight Lights K.S.
- B. Status:
- 1) Three large-scale Semiworks runs using one A/C and three casings were completed on June 19, 1989. (PMSL Blend).
 - 2) Headspace analyses of Eighty-Eight Regular & Lights are complete.
- C. Plans:
- 1) Cigarettes to be produced the week of June 26, 1989.
 - 2) Cigarette analyticals.
 - 3) Subjective evaluations.

PROJECT RING (KOREA) - GOLD/PMSL GOLD

- A. Objective: Development of a Virginia Sweet product for the Korean market targeted toward Pinetree Golden Lights.
- B. Status:
- 1) Flavor development is in progress.
 - 2) Headspace analyses on Pinetree and Pinetree Gold Lights are complete.
- C. Plans:
- 1) Review headspace analyticals.
 - 2) Continue flavor development.

PAN ASIAN MENTHOL - PROJECT CEDAR

- A. Objective: Development of free-standing menthol model to compete with Salem Lights (Hong Kong).

- B. Status: Prototypes were subjectively and analytically evaluated. The PMSL model was subjectively preferred.
- C. Plans: Flavor modifications, if necessary.

MERIT II (HONG KONG)

- A. Objective: Development of a product to be competitive with Kent.
- B. Status: The PMI test was completed and shipped on June 19, 1989.
- C. Plans: Awaiting test results.

MERIT LIGHTS MENTHOL (JAPAN)

- A. Objective: Develop a Merit Lights Menthol for the Japanese market.
- B. Status: Prototypes using various blends and menthol levels are being made. These models will be subjectively screened for further flavor development and modifications.
- C. Plans: Danchi Test scheduled for August 28, 1989.

PROJECT NUMBER: 2307
PROJECT TITLE: Basic Flavor Investigation
PROJECT LEADER: R. W. Hale
PERIOD COVERED: June, 1989

I. ANALYTICAL SUPPORT

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Status:

1. Glycerin/Triacetin: Cigarettes prepared using the natural/synthetic blended triacetin on the filter plugs were subjectively evaluated. All test cigarettes were found to be different than the Celanese synthetic triacetin control.

Method development for in-house triacetin odor evaluation continues.

2. Marlboro Standardization: GC profiles and GC/MS identification of the components have been completed for the following preblends for Marlboro Standardization Run VII:

75-243	75-133
75-197	75-185
75-184	75-605

3. Cambridge: Ethanol, water and propylene glycol were run on five Cambridge aftercut flavors.

4. Trim V (Menthol): GC profiles were run on two concentrates and the finished aftercut.

5. External Analytical Support: The analysis of anethole on 41 filler and 8 aftercut samples has been completed for the aftercut application optimization study in Semi-works.

Ethanol, water and propylene glycol were determined on casings and aftercuts for different projects.

6. Miscellaneous Internal Analytical Support: Analyses were completed for 15 casings and aftercuts for different projects in the Flavor Division.

Humectants and sugars were analyzed by HPLC for 11 samples.

II. BASIC FLAVOR INVESTIGATION:

- A. Objective: To develop basic and applied knowledge for the purpose of flavor improvement or modification of existing products and to provide the basis for development and application of flavor technology for new, unique products.

B. Status:

1. Low Tar/High Flavor: Fractionation of the TPM from 2800 Marlboro 100 cigarettes into the acidic, basic and neutral fractions has been completed. Identification of the components in TPM continues.

PROJECT NUMBER: 4009
PROJECT TITLE: Development Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: June, 1989

I. Reduced Sidestream Cigarettes:

- A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. Status:

Trim V: An evaluation was conducted to determine the effect of O.V. and circumference on sidestream visibility. 17.0mm circumference cigarettes in the Trim V configuration were made at Bryce-Jewett at the correct weight and circumference and at high circumference and low circumference each with reduced weight. The cigarettes were conditioned to different O.V.'s, ranging from 10.9 to 13.5% O.V. Results indicate that as % O.V. decreased, the amount of sidestream smoke increased slightly.

Ultralim cigarettes produced with papers made at the University of Maine containing varying levels of CaCO_3 were evaluated to determine the optimum CaCO_3 content for acceptable sidestream reduction and subjectives. Cigarettes made with 25% CaCO_3 paper showed a 60% reduction in sidestream smoke. Cigarettes made with this paper coated with a 4% solgel solution showed a 78% reduction; however, the cigarettes had an off taste and had an increase in mainstream delivery. Cigarettes made with 45% CaCO_3 showed a 56% sidestream reduction and were preferred subjectively over 25% CaCO_3 cigarettes, possibly due to the 45% CaCO_3 paper being more porous.

Trim V cigarettes were made with narrow innerliners to determine the effect on sidestream reduction of missing or partly missing innerliners. The PDM innerliner was slit to 12.0mm and 14.4mm and was combined with KC's low sidestream outer wrap during Q.E. trials on production machinery in Louisville. The full width innerliner gave 77% sidestream reduction relative to Marlboro Lights 100. The 14.4mm model with the liner partly under the lapseam gave 73% reduction, the 12mm liner gave 69%, and the model with no innerliner gave only 56% reduction.

Lotus: Regular circumference cigarettes were made with papers containing varying levels of CaCO_3 . The model with 45% CaCO_3 was preferred subjectively over one with 25% CaCO_3 by the Studio panel; both models gave a sidestream reduction of less than 50%, and they burned too fast. Cigarettes made with 25, 35, and 45% CaCO_3 paper had increasingly faster static burn times as the carbonate level increased. The 25% CaCO_3 paper was also coated with a 4% solution of Alumina solgel; cigarettes made with this paper had a puff count 1.5 puffs higher than non-coated cigarettes and had a slight off-taste.

Models were made with papers produced at the University of Maine containing several alternative inorganic fillers; all were sized with standard levels of potassium succinate, CMC, and MAP. Cigarettes with a $\text{MgCO}_3/\text{CaCO}_3$ mixture filler were not universally accepted subjectively. Cigarettes with a MgCO_3 filler were preferred over a control KC single-wrap low sidestream paper, and showed a sidestream reduction of 60% over Marlboro Lights 100. Cigarettes made with a hydrotalcite filler were acceptable subjectively and gave a 60% reduction on sidestream smoke.

II. Sidestream Visibility Measurements:

- A. **Objective:** Determine sidestream visibility of experimental models and commercial brands.
- B. **Status:** The eight-port visibility instrument has been qualified by U.S. Testing. The second round of qualification burns consisted of five replications each of Marlboro Lights 100, a very high sidestream model, Capri, Trim V, and a 50/50 mix of Marlboro Lights 100 and the lowest sidestream regular circumference model. Runs were also made using six, four, and two Marlboro Lights 100 with the remaining holders empty to determine the linearity of the measurements.

The sidestream visibility of a series of low-density cigarettes was measured on the eight-port visibility instrument. These models were designed to give the same tar and puff count as control cigarettes. Solgel-coated paper and KC's low sidestream paper were used in the evaluation. Results showed that solgel-coated cigarettes with 25% weight reduction showed about a 25% reduction in sidestream compared to control cigarettes.

Burning various handmade cigarettes on the single-port showed the following results: a model with vanilla-hydrotalcite and multiflex mm as filler in the paper gave a 27% sidestream reduction; there was no difference in sidestream between models coated with solgel at 10, 20, and 30% of the CaCO_3 filler; the alkoxide version of solgel from N.Y. Poly was more effective than the inorganic version in reducing sidestream smoke; cigarettes made with solgel slurry-coated paper burned slower and gave better sidestream reduction than cigarettes made with solgel paste-coated paper; solgel made at pH 10 gave slightly better sidestream reduction than solgel made at pH 10.5.

PROJECT NUMBER: 2304, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: G. N. Yatrakis, J. B. Easley, and J. L. Spruill
PERIOD COVERED: June, 1989

I. LOW TAR/HIGH FLAVOR:

A. Marlboro Ultra Lights

1. Objective: Develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.
2. Status: Production specifications revised to include analytical data for all four packings produced at Cabarrus in May. A change in total RTD target for the 100mm specifications was included.
3. Plans: Monitor test market samples.

B. Project 605

1. Objective: Develop a 6mg free-standing cigarette which appeals to flavor low smokers.
2. Status: CA and CA/CA-Web models have completed internal testing. The concentric peripheral flow/CA models have completed analytical and are being subjectively evaluated. Selected models will be tested in-house.
3. Plans: Complete subjective evaluations and test internally.

C. Project 202

1. Objectives: Develop a 2mg product subjectively comparable to Merit Ultra Lights.
2. Status: Blend 3 models have been made using two new total blend casings. Subjective evaluations are in progress.
3. Plans: Selected casing model will be used for aftercut and filter development.

D. Novent

1. Objective: Develop a 6 mg tar cigarette without filter ventilation.
2. Status: Two models (CA/CA-Web and a concentric peripheral flow CA) have been selected for additional analytical and visible sidestream analysis. Prototypes have been completed (Blend A and 100% XTH blend) for analytical base lines for blend development.
3. Plans: Blend, flavor system and filter development.

II. TRIM I:

- A. Objective: To develop ultraslim product candidates with 17mm circumference that demonstrate product advantages over Capri and Capri Menthol.
- B. Status: Blend re-evaluation in progress.
- C. Plans: Casing and aftercut development.

III. LOW SIDESTREAM:A. Trim V

- 1. Objectives: To develop ultraslim product candidates with 17mm circumference with reduced visible sidestream that demonstrate product advantages over Capri and Capri Menthol.
- 2. Status: POL's 7185 and 9089 are complete. POL's 7186 and 9090 have been shipped. Three makers and packers installed in Louisville. Factory trial was conducted in Louisville for the menthol version. Menthol salesman samples production began 6/26/89.
- 3. Plans: Monitor factory production of salesman samples.

IV. CARTIER:

- A. Objectives: To develop a Cartier similar to Europe's product for the U.S. market.
- B. Status: Consumer testing in progress in Dallas and Kansas City evaluating Famous 2A and 2B and Virginia Slims Lights in 100mm configurations. Analytical is complete and subjective evaluation is in progress for the mentholated prototypes.
- C. Plans: Complete Mall Placement testing.

V. PARLIAMENT LIGHTS L.S. FTB MENTHOL:

- A. Objectives: To develop a Parliament Lights Long Size Flip Top Box Menthol product acceptable to the U.S. Market.
- B. Status: Ad/pack results due week of July 3rd. Twenty-one day spotting evaluation showed no difference compared to 5 day spotting.
- C. Plans: Test Market to be decided.

VI. PROJECT TOMORROW:

- A. Objective: Develop improved product candidates utilizing materials, designs and technologies currently under development.
- B. Status: Remake of POL's 3617 & 3618 (Marlboro vs. Tomorrow Blend N) is complete and awaiting analyticals. Phase III LDR model has been retested on the M.C. Panel and showed no significant difference when compared to the control Marlboro. Phase II using K and L blends will be retested on the M.C. Panel.
- C. Plans: Release POL's pending analytical and subjective evaluation.

VII. PROJECT AMBROSIA:

- A. Objective: Develop a 23.0mm circumference aromatic sidestream product and apply this technology to other products.
- B. Status: Experimental adhesive samples have been received from National Starch incorporating Aromatek 240 & 245 at 3.5% loading. Adhesives are currently being evaluated for maker speed performance and subjectives. Pilot scale quantity of Aromatek 245 has been ordered and is due week of July 3rd.
- C. Plans: Continue to optimize our paper application technique.

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PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development-Asia/Pacific Region
PERIOD COVERED: June, 1989
WRITTEN BY: C.B. Altizer

I. PROJECT RING (Korea)

- A. Objective: Develop new products to compete with 88 Lights and Pine Tree Golden Lights.
- B. Results: Prototypes to compete with both products are ready for consumer evaluation.
- C. Plans: PMAI has requested that Seoul Consumer Panel Test #3 SCP evaluate our 88 Lights prototype (PMSL filter). Test #3 should be ready to ship by 9/1/89. Parliament prototypes for the test will be made at Stockton Street.

II. PROJECT SINGAPORE (Singapore)

- A. Objective: Ensure PM brand line-up conforms to the impending Tar/Nicotine Legislation which requires all brands to yield a maximum of 15.0 mg. Tar and 1.3 mg. Nicotine as measured by the UK method. All of our export products manufactured after 8/1/89 must conform. PMAI has requested that even our "Duty Free" products, sold to Singapore, comply with the legislation.
- B. Results: Prototypes have been made on all products which did not conform to the new law, except Lark KS SP, and Marlboro Menthol. Specifications are being written for all revised products.
- C. Plans: A factory trial was completed in Louisville 6/30/89 on Marlboro Menthol (non-diluted). A factory trail at the M/C will be performed on Lark KS SP after factory shut-down. As specifications are completed and approved, they will be provided to Operations Services.

PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PROJECT LEADER: S. B. Nelson
PERIOD COVERED: June, 1989

I. LARK 1989 PROGRAM

- A. Objective: To optimize the subjective acceptance of the Lark family while retaining the Lark character.
- B. Status: Top-line results of the following two Lark Super Lights PMI Tests were reviewed:
- o Current Lark blend versus GLC-3 blend both @ 9.0 mg/cigt. FTC Tar.
 - o Current Lark blend @ 9.0 mg/cigt. FTC Tar versus GLC-3 blend @ 7.0 mg/cigt. FTC Tar

The current Lark and GLC-3 blends at 9.0 mg/cigt. FTC Tar were equally preferred and rated. In the second test the current Lark blend at 9.0 mg/cigt. FTC Tar was directionally preferred and rated over the GLC-3 blend at 7.0 mg/cigt. FTC Tar.

The Danchi test results of the Lark blend/tar evaluations were received and analyzed. The results indicate there was not statistical difference between the current Lark and GLC-3 blends on the liking scale. In addition, there was no difference on the liking scale between Lark at 15.5 and 14.0 mg/cigt. FTC Tar.

II. CHESTERFIELD

- A. Objective: To develop an 11 to 12 mg tar American blended product.
- B. Status: Top-line results of the second PMI test of Chesterfield (GLC-3 Blend with B Flavor System) versus Lucky Strike were received. The results indicated that Lucky Strike was preferred and rated better than the Chesterfield model.

Field work was initiated on a Danchi test to evaluate two alternative blends for Chesterfield. Results are expected the week of July 3rd.

III. ULTRA LIGHTS

- A. Objective: To develop an ultra light product for the Japanese market.
- B. Status: The Danchi test to evaluate two ultra lights models -- Mount blend and "E" at 4.5 mg/cigt. FTC Tar levels were received and analyzed. The two models were equally rated on the liking scale. However the "E" blend was rated as stronger, more irritating than the Mount blend.

IV. PM LIGHTS

- A. **Objective:** To evaluate alternative types of expanded tobaccos for the current blend.
- B. **Status:** The Danchi test to evaluate alternative types of expanded tobaccos -- DIET and BFET -- as a replacement for the current FODET in the PM Lights blend was received and analyzed. There was no difference in liking ratings between the FODET and DIET models. However, the BFET was rated lower and statistically different than FODET and DIET.

V. CARBOWAX REPLACEMENT

- A. **Objective:** To evaluate alternative plasticizers for the replacement of carbowax.
- B. **Status:** The Danchi test results of triacetin replacing carbowax in PM Super Lights were received and analyzed. There was no statistical difference in liking ratings between the control with 6% carbowax and 40 mg carbon and the following models:

#1	3% Triacetin and 40mg carbon
#2	6% Triacetin and 40mg carbon
#3	6% Triacetin and 52mg carbon

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: June, 1989

PACKAGING STUDIES

A. **Objective:** Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Status/Plans:**

Residual Printing Solvents in Packaging Materials: Coordinated evaluation of the following items:

- Next LT KS Men Tipping
- Next LT 100 Men Tpg
- Next ULT KS Men Tpg
- Va Slims Super Slims 100 FT 20
- Next ULT 100 SP 20 (Reprinted)
- Next LT KS SP 20 (Reprinted)
- Decor Merit Free Ultra Low Tar Blue Ink #AOBL 36591 (J&B)
- Water Base Overprint #1121E (CA Corp)
- MUL Reg 64mm Tipping Paper (Lot #90526/CHP/pallet #16...submitted by L/vl for high odor)
- Va Slims Superslims Men 100 FT20
- Marl LS FT 20 (Lead free/Toluol free Red....water base lacquer)
- Va Sl Superslims Reg FT 20
- Va Sl Superslims Men Tpg
- Va Sl Superslims Reg Tpg
- Merit DN ULT KS Men Tpg
- Merit DN ULT KS Reg Tpg
- Merit DN LT KS SP Men 20
- Va Slims Superslims 100 Reg & Men FT 20/200
- 116mm 25# Printed Champagne Foil
- Merit ULT Reg & Men KS SP 20
- Merit LT Reg KS SP 20
- Merit LT Reg & Men KS SP Closures
- Merit ULT Reg & Men KS SP Closures
- Merit Reg & Men KS SP Comp Closures
- Merit LT KS SP 20/200
- Merit LT KS SP Men 20/200
- Merit ULT KS SP 20/200
- Merit ULT KS SP Men 20/200
- National Rosyn 33-1556 and Producer 10-33-9010 adhesives for 40's carriers
- 20/100 Ctn Coupon and Insert
- Cork Tip Brown N-26099-W11 Ink
- Federal 12pt SBS sampled at 5 stages from mill to printing press to evaluate for Methanol

Fiber-Lam, Inc. Shipping Cases: Sample cases for a domestic field test and controlled condition testing are due for delivery the end of this month.

Cartier: Box board material has been changed to James River Gemcote II. 10's plastic end cap evaluation is pending the availability of color matched samples.

Miscellaneous: A change from glassine to polypropylene overwrap on block licorice will be evaluated in Jungle room to simulate summer warehouse storage.

An alternate two-sided tape used to affix cigarette packages to promotional cards at the point of sale was evaluated for Marketing Services.

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: June 1989

I. LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: Fabrication of a redesigned microwave cavity expected to have a higher power capacity and to be less sensitive to process variations is underway. Delivery of the cavity is expected by the end of July. Installation and testing of the cavity is therefore expected to begin in August, dependent upon the sample production schedule.

A problem with frequent jams under the suction tape shoe was traced to an incorrect clearance between the shoe and the suction tape which apparently resulted when a broken shoe was replaced.

Production of samples to support binder application studies by Project 1503 continued.

Preparation of a report documenting the design of the making process was initiated.

- C. Plans: Produce additional samples as requested by New Products for sidestream studies. Produce samples as requested by Project 1503 for binder application studies. Conduct several "baseline" runs to define the firmness/density relationship obtainable with the current process using Marlboro type fillers. Evaluate the impact of moisture addition to the tobacco bed on cigarette quality.

II. Molten Menthol Application to Foil

- A. Objective: Evaluate molten menthol application as a means of applying menthol to foil without the ethanol carrier.
- B. Results: Preliminary trials were conducted to check out the operation of the Kaymich on the PM coater. Several bobbins were produced using the smear roller and have been submitted for testing. Attempts to operate the unit with the foil bypassing the smearing roller were unsuccessful, however, because of interference between the foil and the nozzle bracket. This problem will be alleviated by fabricating a longer mounting shaft for the bracket.
- C. Plans: Fabricate and install a longer nozzle mounting shaft to allow the smearing roller to be bypassed. Determine the effect of residence time on foil contamination. Establish the operating conditions which minimize the retention of menthol on the foil face. Determine the maximum loading of menthol on the foil obtainable with this process.

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PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: June, 1989

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend properties of reconstituted sheet materials.

B. Results:

1. ART Project - Pilot RL incorporating "wax layer" stems from the ART Pilot Plant (the top 2-3% of the stem bed) does not show appreciable subjective differences compared to RL containing the same level of total ART stems. There is increased mouthcoating, but bitterness/aftertaste is reduced. Removing the wax layer of stems would not be effective from a sheet materials subjective standpoint.

Final Marlboro blend subjective results to evaluate ART Pilot Plant stem utilization in sheet materials was completed. Pilot RL containing 3.6% ART stems in the feedstock is detectable in Marlboro blends, whereas 1.8% incorporation in RL appears acceptable. BL Plant RCB containing 22% ART stems is detectable; a psuedo 16% level (a blend of RCB sheets containing 11% and 22% ART stems) is not detectable. The combination of 1.8% in RL and 16% in RCB gave detectable differences; blends with a combination 1.8% ART stems in RL and 11% in RCB appear acceptable.

Stems from the Bermuda Hundred production facility were incorporated in BL Plant RCB at 11% and 16% of the feedstock (replacing burley stems). Screening of handmade 100% cigarettes indicates an improvement versus ART Pilot Plant stems incorporated at the same level; blended cigarettes have been completed and submitted for subjective testing.

Replacing BL Plant burley stems with Bermuda stems will require increased burley stem usage at Park 500 to maintain durations. Pilot RL sheets were made with a range of burley stem contents; screening of handmade 100% cigarettes indicates that Park 500 would be limited to 34% burley stems in the feedstocks (Δ +2.5% over present).

The 34% burley stem blend was used to produce pilot RL sheets incorporating 1.8% and 3.6% Bermuda stems. These have also indicated a subjective improvement (in handmade 100% cigarettes) versus comparable inclusion levels of ART Pilot Plant stems; blended cigarettes are in progress. These same burley stem/Bermuda stem combinations are intended for Park 500 trials, pending acceptable subjective results with the pilot sheets.

Marlboro models containing blend combinations of BL Plant RCB and pilot RL, both made with Bermuda stems, are also in progress. Based on the improved subjective response with Bermuda stem incorporation, this test grid is being expanded to attempt higher overall blend utilization levels than were indicated as feasible with ART Pilot Plant stem incorporation.

Bermuda filler was incorporated as 5% of RCB feedstock (replacing CTRL) to evaluate this as an outlet for Bermuda and MC ART OTM. There were no subjective differences detectable in handmade 100% cigarettes; evaluation of machine-made blended cigarettes is in progress. Bermuda filler has also been incorporated in pilot RL at 5% and 10% of the feedstock (replacing scraps) to determine any subjective effects.

2. Humectants - Cigarettes were made to determine the optimum isosweet level for replacement of humectants in the strip casings for PG/glycerine-free blends, and have been submitted for analyticals and subjective screening.

C. Plans:

1. Complete blended cigarette screening of pilot RL and BL Plant RCB sheets made with Bermuda stems.
2. Conduct Park 500 trials incorporating Bermuda stems in RL.

II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. Liquid Flavors - Larger quantities (suitable for plant trials) of the Takasago flavor were received and subjectively screened (sprayed on pilot 150B made without dry flavors). The Takasago materials continue to be analytically and subjectively consistent, and are subjectively preferred. Chart has corrected their roasting problem and reserved a trial quantity lot; a sample was screened and judged comparable to past "acceptable" Chart flavors. The large lots from both vendors are intended for compounding (sugar adjustment) in the Flavor Center and evaluation in pilot 150B. Combinations of the two flavors will subsequently be evaluated; selected combinations will be used for sheet plant trials. Flavor Development plans to visit both vendors in July and provide them with flavor specifications.

C. Plans:

1. Produce pilot 150B sheets to evaluate compounded liquid flavors.

PROJECT NUMBER: 1308
PROJECT TITLE: Papermaking Process Development
PROJECT LEADER: R. M. Rogers
PERIOD COVERED: June, 1989

I. HANDSHEET PRODUCTION

- A. Objective: Develop proprietary cigarette papers for low sidestream and other new product applications.
- B. Results: Handsheets were produced to evaluate precipitated sol-gels formed via the organic process. Calcium phosphate gel precipitate exhibits sheet making properties similar to other fillers formed via the organic process. Poor sheet drainage limits usage levels. Sheets containing 10% gel and 20% calcium carbonate exhibit low porosity levels with extremely poor brightness and opacity. However, both the alumina coated calcium carbonate and calcium carbonate gel precipitates exhibit superior sheet making properties similar to commercial calcium carbonate. Brightness, opacity and porosity response to freeness are acceptable. Sidestream smoke evaluations are in progress.

The addition of potassium salts as burning additives appears to substantially reduce sidestream smoke for sheets containing a number of different fillers. Nominal levels of potassium, up to 1.5%, appear to increase burn rate and reduce sidestream smoke. Higher levels decrease burn rate with a further reduction in sidestream smoke. Side stream smoke performance may also be a function of the type of potassium compound. Since the burn rate is also influenced by sheet porosity, the effective potassium level could be matched with the porosity required to achieve acceptable subjectives, possibly with conventional porosity cigarette papers.

The capability to coat water insoluble particle suspensions, such as calcium carbonate or titanium dioxide, on cigarette paper handsheets has been developed. The coating is evenly distributed across and down the sheet with acceptable appearance. This methodology is similar to the operation of either a reverse or forward roll coater.

A variety of different handsheets were produced this month incorporating various fillers, including potassium magnesium phosphate. In addition several carbonate sheets were sized with various potassium compounds in an attempt to alter not only sidestream smoke but also subjectives. The dual wrap concept is being utilized by combining radically different sheets such as low pH inner wraps and alkaline filled outer wraps. Thus the negative subjective character of highly sized sheets, required for sidestream smoke performance, can be isolated by incorporating a porous inner wrap or possibly altered by the inclusion of an acid treated inner wrap.

C. Plans:

1. Continue the preparation of handsheets to evaluate designed fillers and sol-gel preparations.
2. Evaluate the application of potassium compounds to conventional cigarette papers.
3. Investigate the addition of mixes of various fillers and adhesives (sol-gel) to cigarette papers to control sidestream smoke and/or mass burn rate.

II. PILOT PRODUCTION

- A. Objective:** Produce pilot quantities (bobbins) of cigarette paper.
- B. Results:** A series of ten sheets are being produced at Maine in support of the low sidestream smoke project. Four of the sheets are light weight, low filler sheets utilizing calcium carbonate and magnesium carbonate for use as the inner wrap of a dual wrapped cigarette. Two porosity levels were chosen, 13 and 50+ Coresta. The more porous sheet is definitely at the processing limit of the type A flax currently being used. If more porous sheets are required, given the same specifications, another fiber must be used.

Additional refining runs confirmed the difficulty of refining the current shipment of Type A flax. The options available to improve sheet appearance and produce a high porosity paper are limited. Refining levels required to achieve satisfactory appearance produce sheets with low porosity. Other grades of flax or woodpulp blends produce sheets with higher porosity levels and superior appearance. These options are being investigated.

A set of bilayer sheets are being produced to continue evaluations of outer wraps with magnesium-containing compounds and inner wraps with magnesium carbonate. Certain magnesium-containing fillers appear to reduce sidestream smoke delivery but retain substantial negative subjective attributes. The inner layer of magnesium carbonate may alleviate this problem.

A vergeure roll and a stock screen for the secondary headbox at the University of Maine have been installed. These units will be operated during production of bilayer sheets at the end of July.

C. Plans:

1. Complete production of low filler inner wraps, bilayers and higher weight single sheets.
2. Modify both the vergeure roll and the new stock screen as required to achieve acceptable performance.

PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: June 1989

I. LOW DENSITY ROD - BINDER APPLICATION

- A. Objective: Produce precoated filler via the batch or continuous process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.
- B. Results: The effect of pectin solution concentration on cigarette firmness was tested. Using a 2% solution at a 6% application level, a 3.6 mm firmness was achieved at a 25% tobacco weight reduction. A 6% solution at a 5.4% application rate yielded a 3.6 mm firmness. It appears that firmness is more dependent on application rate than concentration.

Approximately 600 pounds of filler were received from Australia. The material is being coated for cigarette production.

- C. Plans: Experimentation is being conducted to determine factors affecting cigarette rod firmness.

II. BINDER TECHNOLOGY

- A. Objective: Investigate the mechanism of filler bonding and stiffness produced by coating. Improve commercial feasibility by examining alternate binders and processes.
- B. Results: Alternate binders, potato starch, and potato derived maltodextrin solutions were used to coat Marlboro filler. A series of solution concentrations and applications levels was evaluated. Cigarettes were produced on the Low Density Rod maker and submitted for firmness and subjective testing. Visually, some of the samples had satisfactory bonding of the filler.

Yucca was re-evaluated as a surfactant for use with pectin. Filler was coated and cigarettes were produced. Samples are currently being tested.

- C. Plans: Reduced density rods produced on the maker will be reactivated a second time in the laboratory steam chamber to examine if firmness can be improved.

PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: June, 1989

I. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. **Objective:** To conduct trials providing information for development of the ART commercial process.
- B. **Results:** To investigate the cigarette spotting issue, the operation of the Bermuda Hundred Hauni tunnel on the extracted filler line was examined to determine its performance relative to the Semiworks. Upon inspection, BH engineers discovered, and immediately corrected, a faulty solenoid on the tunnel condensate line. In addition, it was found that the distribution of filler to the BH tunnel was somewhat less uniform than in the Semiworks, that the filler residence time in the BH tunnel was less than in the Semiworks tunnel, and that the steam to filler ratio was lower at BH. To date, the residence time has been increased to approximately 30 seconds by installing a variable speed drive, and additional holes have been drilled in the BH tunnel to increase the steam to filler ratio. Also, the uniformity of the feed to the tunnel has been significantly improved.

As the tunnel modifications were being made, several Semiworks trials were conducted with Bermuda Hundred filler to verify that the extraction step was causing the spotting and to compare tunnel processing at the two locations. Cigarettes made from fillers sampled immediately before and after extraction yielded SOLVIC spot values of 0 and 123 critical spots/2000 cig, respectively, demonstrating that the extraction step is producing the spotting potential. Semiworks trials were run producing finished filler, with and without tunnel treatment, from extracted material. These showed significant reductions in spot values for the tunnel treated models to levels comparable with past Pilot Plant results. All of the above testing was conducted on non-menthol models.

To address the menthol models, Bermuda Hundred filler was obtained for cigarette making and packaging in materials which targeted menthol loadings of 0, 86, and 100 mg/pack. SOLVIC analysis showed dramatic increases in the spotting levels for the latter two configurations.

In addition, testing of menthol models was conducted after the above tunnel modifications were completed. Finished filler was obtained from BH as was extracted filler that was processed into a final blend at the Semiworks, with and without tunnel treatment. Cigarette inspection (5 day aging) showed values of 94 critical spots/2000 cig for the BH filler, and 93 and 133 critical spots/2000 cig with and without Semiworks tunnel treatment.

As part of the program examining alternatives for expansion of extracted filler, the one liter facility conducted extraction runs

on DIET, expanded DL blend, and a blend of 80% unexpanded DL/20% expanded DL to determine if any cylinder volume collapse occurred during extraction. Each material was run at two AB/OV target levels, 3.0%/26% and 1.3%/19%, and was sampled after AB application and after extraction. The CV (cc/g) values were:

	<u>DIET</u>	<u>Exp. DL</u>	<u>80% Unexp. DL/ 20% Exp. DL</u>
starting material	8.5	9.2	5.9
high AB/OV, cased	8.2	7.8	5.9
high AB/OV, extracted	6.5	6.8	5.6
low AB/OV, cased	8.3	7.7	6.0
low AB/OV, extracted	7.0	7.4	6.0

- C. **Plans:** Additional spotting analyses are being run comparing cigarettes made from filler processed through the BH tunnel versus the Semiworks tunnel. Also, trials have been conducted with BH filler in the Semiworks operating the tunnel at different residence times and tobacco throughputs. Spot data will be available next month.

To test the extraction of expanded materials on a larger scale, the Bermuda Hundred Pilot Plant made runs with the 80/20 blend and the 100% expanded DL. Data generation and analysis are continuing.

II. HAUNI HT TUNNEL EVALUATION

- A. **Objective:** Determine the effect of steam conditioning tobacco materials in the Hauni HT steam tunnel prior to drying.
- B. **Results:** The fourth (and final) test in the initial evaluation series was conducted processing Marlboro cut rag through the Semiworks Hauni tunnel to determine the effect of steam pressure on cigarette firmness. However, firmness testing has been delayed due to other priorities in the laboratory.
- C. **Plans:** Complete physical and subjective evaluations on all four tests.

III. TMCi-ASTA SHEET

- A. **Objective:** To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCi process and PM-RCB technology for international application.
- B. **Results:** The Cadiz plant produced ASTA on one line in June. The plant will be shut down in July for rectification of problems on this line and completion of the second line. ASTA product from Cadiz has the same pinholed appearance as the Tarragona product from the same tobacco grind (120 mesh) and PM binder formulation. Doctoring of the sheet from the belt appeared to be acceptable as

judged from the appearance of the underside of the sheet. The sheet dryer capacity has been reported by TSA to be only 50% of production capacity when casting slurry at 19% slurry solids (design 21%). However, there are still significant problems with the rub rails on the dryer steam boxes which are limiting factors in assessing the potential dryer capacity. A questionnaire was sent to TMCI in Cadiz to establish the status of outstanding mechanical questions and the quality of the process operation.

The measurement of feedstock bulk densities and drying rates of ASTA sheet made from various blends and grind sizes did not show major differences. This information was sent to TSA and TMCI who were concerned that blend changes might influence the performance of the Cadiz plant.

Air removal from ASTA slurry in the laboratory was confirmed to minimize pinholes in the sheet. Tensile strength, elongation and work to break values for the product from which air had been removed were increased by 30-50% for both ASTA and RCB. The density of the sheet was also increased by air removal. The ammonia content of the slurry was not measurably changed by the application of vacuum to the slurry to reduce its air content. Commercial equipment is available for rent to confirm these observations on a production line.

The measurement of survivability of ASTA filler from laboratory handsheets by sieve analysis was found to be unrepresentative for filler from the finest tobacco grind. This product has a very smooth plastic appearance and the filler which was ~1" long passed through the 6 and 12 mesh sieves. Another means of measurement of filler length is being sought.

- C. Plans: Discuss a production trial with BL Plant management for air removal from RCB slurry to improve sheet quality.

Support TSA in future Cadiz trials.

Develop an alternative means to sieve analysis of quantifying filler size of reconstituted tobacco.

PROJECT NUMBER: 1810
PROJECT TITLE: ART Process Development
PROJECT LEADER: Ravi Prasad
PERIOD COVERED: June, 1989

I. PROJECT ART - COMMERCIAL PLANT

- A. Objective: To support the Bermuda Hundred Processing Facility startup/operation.
- B. Results: R&D support to the commercial plant startup was provided via the assignment of pilot plant personnel for round-the-clock coverage. The process feasibility of achieving 97% nicotine extraction was established. The remaining issues with the commercial plant are 1) to consistently load the filler and stems into the vessels in a uniform fashion and, 2) to modify the CO₂ "fill" sequence to avoid disturbing the bed.

After a successful startup, R&D personnel returned in mid-June to operate the Bermuda Hundred Pilot Plant. The commercial plant continues to be supported by R&D for data analysis, special sampling, and process optimization.

The delivery of Hinds on-line monitor for the commercial plant has been delayed. R&D support will be provided when this unit is received and installed by commercial plant personnel.

- C. Plans: Continue the support of BHPP, on an as needed basis.

II. PROJECT ART - BHPP

- A. Objective: To support commercial plant design and flavor development objectives at the Bermuda Hundred Pilot Plant.
- B. Results: The Bermuda Hundred Pilot Plant was started up on June 19, 1989. After initial pressure testing, three control runs were completed, using filler and stems from the commercial plant, to verify that the target levels of extraction can be achieved.

Two expanded tobacco runs were successfully completed (100% DIET; 80% DL Blend/20% DIET), giving target levels of nicotine removal. Results of CV gain/loss will be reported separately.

A series of extraction runs, using citric acid on stems, was initiated for product quality improvement. Corrosion will be monitored with the help of several corrosion coupons and with the analysis of condensate drain samples.

Construction of the BHPP office addition has been completed.

A 650 for cooling heat exchanger was approved. The heat exchanger will allow optimization of the current stem absorption process and it will also allow moisture balance of the filler when a liquid absorber is used to remove nicotine from CO₂.

C. Plans:

1. Continue the evaluation of citric acid on stem absorber, leading to improved filler subjectives.
2. Complete the feasibility of extracting nicotine from expanded filler.

III. PROJECT ART - NEW PROCESS DEVELOPMENT

- A. Objective:** To develop new processes for scale-up and implementation in the Bermuda Hundred Facility.
- B. Results:** Further meetings were held with Air Products and Chemicals, Inc. to define an oxygen scavenging system in an effort to reduce the potential for chloride stress corrosion cracking in 316L stainless steel.

Working vessel drawings and CO_2 flow information were forwarded to Air Products. This information will be used by Air Products to formalize a process concept for removing O_2 from CO_2 via oxygen scavengers.

The water column flooding velocity tests at General Foods Hoboken were rescheduled for the week of June 19, 1989. Testing is currently in progress and data will be reported upon completion.

Liquid absorber design and development timetable is being reviewed with Engineering in the context of capacity expansion scenario for the Bermuda Hundred Plant. A 650 for batch liquid column was approved, and the basket assembly will be ordered by July 14, 1989.

C. Plans:

1. Continue the process design of oxygen scavenging system for BHPP, to the point of P&ID and 650 preparation.
2. Accelerate the design activity for the liquid absorber concept, in cooperation with Engineering and outside consultants.

PROJECT NUMBER: 1811
PROJECT TITLE: Process Chemistry Development
PROJECT LEADER: S. E. Wrenn
PERIOD COVERED: June, 1989

I. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. Objective: To conduct experimentation providing information for development of the ART commercial process.
- B. Results: A laboratory spray system was assembled to determine factors relating to the clogging of the nozzles of the AB cylinder at Bermuda Hundred processing plant. Several factors were identified as contributors to the clogging problem including a solution which is very close to the solubility level, a temperature drop at the nozzle, and degassing of the solution in the lines and at the nozzle allowing crystal growth to begin. In the lab spray unit, the crystal formation could be eliminated by increasing the velocity of the spray at the nozzle. It could also be eliminated by heating the nozzle or lowering the concentration of AB in solution. When the nozzles were heated, degassing at the nozzle increased resulting in a more sporadic spray. The majority of the work was done using an internal mix nozzle. When an external mix nozzle was used, no clogging of the nozzle occurred. However, crystal growth was observed in the fluid cap of the nozzle.
- C. Plans: Discuss lab results with Bermuda Hundred personnel. Continue to assist the commercial operation as needed.

II. PROJECT ART - Liquid Tower Studies

- A. Objective: To conduct experimentation to provide information for the development of a liquid column concept to remove nicotine from supercritical CO₂ in support of the pilot plant operation.
- B. Results: A model of a wedge section of a basket for the liquid tower was constructed in the lab. A light mineral oil with a density of 0.8 g/ml, interfacial tension of ~5 dynes/cm, and a viscosity of 16 cps was used to represent CO₂. Flow patterns of the fluid indicated that a baffle at the CO₂ input was causing a region of high velocity on the plate. By removing this plate, the flow was more evenly distributed across the plate wetting all the available holes. As the oil enters the vessel covering the plate, a dead region of water under the plate is made. The thickness of this region is determined by the rate at which the oil is added to the vessel.
- C. Plans: Experiment with increasing velocity and lower viscosity fluids. Change configuration of vessel with addition of baffles to bottom of vessel.

PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: L. Ryan
PERIOD COVERED: June, 1989

I. METHOPRENE

- A. Objective: To determine the most efficient use of the insect growth regulator, methoprene within PM's cigarette beetle (CB) management program.
- B. Results: A study to determine potential methoprene deposition on tobacco during Dianex® applications (1) was completed. This involved 4 replicate Dianex® aerosol applications in D Pilot Plant and the sampling of bright cut filler (CF) at various stages of simulated CF production. Samples of CF were analyzed by ARD personnel using HPLC techniques (2). No residues of methoprene were detected at any time on the production CF.

A document describing the possibility of using ELISA techniques to detect methoprene at <1 ng levels was received (3) and discussed with interested parties at R&D.

- C. Plans: Evaluate the CF samples for methoprene using CB bioassay techniques and for potential xylene residues. Submit a special report on the study to date. Continue to assist in the integration of methoprene into PM USA's integrated pest management program.

D. Reference:

1. Ryan, L. and Lehman, R. M. Proposal: To Study the Potential Methoprene Deposition on Tobacco During Dianex® Applications. Memo to Faustini, D. L. May 22, 1989.
2. Yang, S.-S. Notebook No. 8778 pp. 55-63.
3. Yin, C.-M. of the University of Massachusetts. ELISA for Methoprene Detection. Letter to Lehman, R. M. May 1, 1989.

II. FUMIGATION

- A. Objective: Investigate alternatives to conventional fumigants for the disinfestation of bulk tobaccos.
- B. Results: Evaluation of vacuum-steam conditioning as an alternative to methyl bromide for the fumigation of export cut filler (ECF) to obtain phytosanitary certificates (phyto) is underway (1,2). The relevance of the phyto to PM USA's ECF business is being investigated (3).

- C. Plans: Gain management approval for research studies aimed at gaining phytos based on probe-equipped vacuum-steam conditioning cycles and assist, as requested, with efforts to move ECF production to Stockton Street.
- D. Reference:
1. Ryan, L. and Faustini, D. L. Vacuum-steam Conditioning as an Alternative to Methyl Bromide Fumigation of Export Cut Filler to Obtain Phytosanitary Certification. Memo to Distribution. June 5, 1989.
 2. Houghton, K. S. R&D's Effort Regarding the Concerns for Cut Filler Fumigation. Memo to Golay, A. May 23, 1989.
 3. Ryan, L. Phytosanitary Certificates (Phyto) for Export Cut Filler (ECF). Memo to Charles, V. May 25, 1989.

III. SERVICE TO OTHERS

- A. Objective: To provide technical services to areas outside R&D.
- B. Results: Assistance was given in the following areas:
- moisturizing cylinder CB efficacy tests as part of the Bulk Tobacco Handling (BTH) program at the RLPH (1,2), O/C Semi-works (3) and for start-up at Bermuda 100 (4).
 - vacuum-steam conditioning CB efficacy tests as part of an engineering project to enhance the safety of conditioning units (5).
- Advice was given on sanitation and pest control at the Cabarrus manufacturing facility (6) and the visit from PME's sanitarian was completed (7,8).
- C. Plans: Continue to gather data, within the BTH program, on the relationship between temperature of and residence time within moisturizing cylinders and CB mortality.
- D. References:
1. Ryan, L. and Lehman, R. Cigarette Beetle Mortality in a Moisturizing Cylinder at the Richmond Leaf Processing Facility. Memo to Rowe, N. R. May 25, 1989.
 2. Ryan, L. Cigarette Beetle Mortality in a Moisturizing Cylinder at the Richmond Leaf Processing Facility. Memo to Grove, M. A. June 1, 1989.
 3. Lehman, R. M. and Ryan, L. Cigarette Beetle Mortality in an Operations Center (OC) Semi-works Moisturizing Cylinder. Memo to Rowe, N. R. June 2, 1989.

4. Ryan, L. Cigarette Beetle Mortality in a Stem Conditioning Cylinder at Bermuda 100. Memo to Werkmeister, P. May 25, 1989.
5. Ryan, L. and Lehman, R. M. Cigarette Beetle Mortality in a Mohr Vacuum-steam Conditioning Unit at the Richmond Manufacturing Center. Memo to Cooper, R. N. May 25, 1989.
6. Faustini, D. L. Sanitation and Pest Control Review - Cabarrus, N. C. Memo to McCuen, R. W. May 18, 1989.
7. Ryan, L. and Faustini, D. L. Visit to Cabarrus by J.-M. Freymond. Memo to Sexton, M. A. and Bridges, C. T. May 8, 1989.
8. Ryan, L. and Faustini, D. L. Letter to Yehman, H. of Degesch America, Inc. April 25, 1989.

IV. OTHER

A variety of meetings were attended by Project 1101 personnel to keep abreast of developments in the fields of insect communication (1,2) and pesticides (3,4).

References:

1. Ryan, L. The Powell Lecture - Summary. Memo to Faustini, D. L. May 25, 1989.
2. Faustini, D. L. 1989 Annual Banquet - ESA Washington, D. C. Memo to McCuen, R. W. May 15, 1989.
3. Minor, M. F. The National Research Conference, Pesticides in Terrestrial and Aquatic Environment - TRIP REPORT. Memo to Faustini, D. L. May 30, 1989.
4. Faustini, D. L. Tobacco Pesticide Committee (TPC) of the Tobacco Workers Conference. Memo to McCuen, R. W. May 15, 1989.

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: June, 1989

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

- A. Objective: To develop methods to objectively and reliably evaluate human responses to smoke constituents and tobacco flavorants.

B. Results:

Cognitive NERP Study

Cognitive NERP experiments aimed at assisting Flavor Development evaluate various sources of natural menthol and synthetic menthol substitutes for natural menthol have resumed. Efforts are currently focusing on the evaluation of two alternative natural menthol sources as compared to the current source.¹

- C. Plans: Continue NERP testing of various synthetic and natural menthol substitutes for Flavor Development. Complete testing of ten subjects in the 75:25 synthetic/natural menthol mixture vs synthetic menthol comparison.

D. Reference:

1. Martin, B. Notebook No. 8689, pp. 170-176.

PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: June, 1989

I. AEROSOL GENERATION AND MEASUREMENT

- A. Objective: Develop the capability for accurately measuring the refractive index of liquids over the wavelength range employed in the light extinction spectrometer.
- B. Results: The Metricon refractometer has been received and found to provide an accuracy of .001 for a variety of test liquids. The refractive index obtained for the cigarette smoke condensate is tabulated below.

<u>Wavelength (nm)</u>	<u>Refractive Index (30°C)</u>
632.8	1.520
611.9	1.522
594.1	1.524
543.0	1.529

- C. Plans: Begin characterization of the aerosol generator incorporating a porous tube.

II. INDIVIDUAL CIGARETTE INSPECTION

- A. Objective: Develop methods for the on-line inspection of individual cigarettes.
- B. Results: The custom designed acoustooptic laser scanning system has been delivered. The system will be used for rapid scanning of cigarettes while being turned on a Hauni roll drum. The equipment was taken to Brimose for checking and improved calibration as well as personal training in its operation. The system now seems to be operating properly.
- C. Plans: Develop a method for accounting for the loss in diffraction efficiency with distance of incident light from the transducer.

III. PACK INSPECTION

- A. Objective: Develop and implement a method for the on-line inspection of cigarette packs.
- B. Results: Itran Corporation has submitted a proposal for the modification of an Itran CI-750 vision system to enable it to execute the PM inspection algorithm. This would facilitate the factory evaluation of the algorithm. However, because of the lengthy development time proposed (7-8 months), the factory

evaluation will instead be carried out using the laboratory inspection system (Sun workstation + Androx imaging board). Engineering has agreed to support the factory evaluation at Stockton Street with a target date of October 1 for the start of the evaluation.

The adaptive method for generating the inspection filter was originally developed with the very high speed print inspection problem in mind. However, it also offers several advantages for the pack inspection problem. It makes it easier to consider large training sets and both reduces the time and simplifies the hardware needed for the training step. Training and inspection simulations were carried out to determine the optimum value of the learning rate parameter.

A large set of video images of Lark packs was obtained earlier while being transported on Engineering's prototype vacuum conveyor. The images have now been digitized and corrected for lighting variation. Inspection simulations using these images have been initiated.

- C. Plans: Incorporate the adaptive algorithm and the new centering and lighting correction routines in the software for the laboratory inspection system. Complete inspection simulations on the Lark image library.

IV. INSPECTION OF INCOMING MATERIALS

- A. Objective: Develop and implement a system for the off-line QA inspection of cut closure stamps and packaging blanks.
- B. Results: The inspection algorithm used in the laboratory closure stamp inspection system has been enhanced in two ways. First, time dependent fluctuations in lighting level are now taken into account. Second, an alternate algorithm based on an edge-enhanced image has been incorporated into the inspection. The new algorithm has advantages in the inspection of white stamps with limited print.

The design of the camera mount for the package blank inspector has been altered to reduce its sensitivity to vibration. The additional machine work has been completed.

- C. Plans: Evaluate the performance of the stamp inspector using a variety of stamps. Transfer to QA for evaluation. Assemble the package blank presentation mechanism and develop the software needed to control the system and discriminate between test and master blanks.

PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: June, 1989

I. LOW NICOTINE

- A. Objective: Develop second generation processing for ART.
- B. Results: The General Foods facility at Hoboken began pilot trials using their four inch water column to verify the flooding velocity models developed at the University of Texas. Tests are expected to be completed by June 30.

Plans were initiated for installing a second generation absorber in the BHPP. PM engineering was requested to begin efforts for equipment design.

- C. Plans: Work is ongoing.

II. LOW NICOTINE

- A. Objective: Support to ART commercial plant.
- B. Results: Six runs were completed on the one-liter system in order to determine the effects of the ART process on expanded filler. The extracted filler was given to the Tobacco Materials division for further evaluation. Results of the extractions are as follows.

<u>FILLER</u>	<u>FEED OV</u>	<u>FEED NIC (DMT)</u>	<u>FEED AB</u>	<u>% REDUCTION</u>
DIET	26.3%	2.12%	2.9%	95.5%
DIET	18.3%	2.10%	1.4%	91.4%
ET (DL 5)	25.3%	2.62%	3.2%	95.9%
ET (DL 5)	19.0%	2.57%	1.4%	93.5%
80% DL5/20% ET	25.5%	2.80%	2.8%	96.2%
80% DL5/20% ET	18.9%	2.62%	1.3%	94.3%

- C. Plans: Work is ongoing.

PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: June, 1989

I. OPERATIONS SUPPORT (J. Crump and A. Closter in collaboration with the Applied Technology Group)

- A. Objective: Determine the effects of machining on the dynamic viscosity changes that occur in PVA tipping adhesives.
- B. Results: Minor modifications are currently being made to the Mark 10A simulator to facilitate operation. Testing of the effect of roller hardness on the viscoelastic properties of tipping adhesives continues with the Mark 9 simulator. The results to date indicate that the hardness of the rollers does affect the rate of change of viscosity during machining. However, the effects are small and further data is required before definite conclusions can be drawn.

II. MENTHOL STUDIES (T. V. Van Auken)

- A. Objective: Determine the diffusion rate and solubility of menthol in cellulose acetate.
- B. Results: A patent disclosure for loading CA webb with menthol in the absence of triacetin using high moisture environments has been submitted.
- C. Plans: Experiments with high moisture conditions will be continued to determine optimum conditions for loading CA with menthol.

III. LOW DENSITY RODS (S. Ganeriwala)

- A. Objective: Compare the compression properties of low density and control rods.
- B. Results: Compression tests on ART cigarettes relative to Merit controls were completed. The cigarettes were equilibrated to standard conditions prior to testing. The ART cigarettes were found to be slightly firmer than the controls, with the difference being smaller for the menthol models. With nonequilibrated cigarettes, as reported last month, no differences could be resolved.

At the request of Warren Claflin, five brands of cigarettes at three rod weights each were tested after equilibration at standard conditions. The compression tests showed no differences between brands. No resolved differences were seen as a function of rod weight, though the observed trends were in the expected direction. This is probably due to the fact that the total weight variation of 20 milligrams is too small to be resolved by the test.

- C. Plans: This work is ongoing.

PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
WRITTEN BY: K. R. Sanders
PERIOD COVERED: June, 1989

I. LOW SIDESTREAM CIGARETTE PAPERS

- A. Objective:** Examine the ultrastructure of selected cigarette papers and paper additives in support of the low sidestream project.

B. Results:

SEM and EDS were used in characterization of a number of Sol Gels, Chemical Additives and Papers in support of the low sidestream project. These techniques have been found to be invaluable in defining subtle differences in structure and particle size of samples for eventual relation to differences in sidestream delivery and visibility.

Comparisons were made of the gross structures of ash from Marlboro Lights 100's, Capri, Virginia Superslims, cigarettes made with Mg(OH)₂ paper, and cigarettes made with a sol gel coated paper. Ash from the sol gel paper and the Mg(OH)₂ paper maintained their structural integrities similar to their unburned counterparts. They contained what appeared to be "fibers" throughout the ash. The Mg(OH)₂ paper formed a crust with "bubbles" on the surface of the ash. The Capri and Virginia Superslims ash was fluffy and granular containing only trace amounts of "fibers". The Marlboro Lights 100's ash crumbled easily and contained no visible "fiber" structure. Ca and P were in all ash samples, and Al was also found in the sol gel ash.

C. References:

1. Yi, G., "Structural and Elemental Characterization of Pulverized Sol Gel," Memo to A. Kallianos, 30 May 1989.
2. Sanders, K., "Characterization of Sol-gel and Reheis Alumina Samples," Memo to A. Kallianos, 13 June 1989.
3. Yi, G., "Elemental and Structural Characterization of MgAl₂O₄ Powder," Memo to A. Kallianos, 18 June 1989.
4. Baliga, V., and Henry, B., "Characterization of Two Magnesium Phosphates and Two Aluminum Phosphates," Memo to J. Paine, 11 May 1989.
5. Sanders, K., "Characterization of CaCO₃ Samples," Memo to K. Podraza 15 June 1989.
6. Sanders, K., "Characterization of CaCO₃: Comparison of Samples Ground and Not Ground," Memo to K. Podraza 19 June 1989.
7. Sanders, K., "Characterization of Al₂O₃/Fe₂O₃ Sample," Memo to J. Fournier 21 June 1989.
8. Yi, G., "Structural and Elemental Characterization of Cigarette Paper Ash," Memo to F. Del Valle, 18 May 1989.

9. Yi, G., "Structural and Elemental Characterization of Experimental Paper Ash," Memo to S. Baldwin, 14 June 1989.

II. ART

- A. Objective: Examine samples for the continuing research and development efforts of the ART Pilot Plant.

B. Results:

Stem widths were measured from three samples of stems which were cut at 150 cuts per inch (0.167mm) then dried and equilibrated. The mean values were; Bermuda 100, $0.33 \pm 0.13\text{mm}$; Bermuda 100 exit Cutter #1, $0.27 \pm 0.1\text{mm}$; Louisville, $0.28 \pm 0.1\text{mm}^1$.

Three samples of DL-5 ART shreds were examined to determine if there were any gross morphological differences between the samples as a result of added sprays. One had water sprayed on, the second had ammonium hydroxide, and the third had ammonium bicarbonate. In all samples, the normal tobacco morphology was maintained. No distinguishing crystalline structures were noted on the surfaces.

C. References:

1. Henry, B., and Baliga, V., "Comparison of Cut Stem Widths from Bermuda 100 and Louisville," Memo to J. Baggett, 6 June 1989.
2. Sanders, K., "Characterization of ART Lamina," Memo to R. Prasad, 16 June 1989.

III. SUPPORT TO OPERATIONS

- A. Objective: Examine samples for support to operations.

B. Results:

A stainless steel plate used as part of the machinery of the RCB casting process was received for documentation of a coating that was visible on one of its surfaces. The coating provided a smooth surface over the plate. It was comprised of particles $\leq 0.2\mu$ in size, and contained primarily Ca with P and Si. Particles $> 0.2\mu$ in size which were not a part of the coating were noted on the surface. These contained Na, Mg, Al, Si, P, S, Cl, K, and Ca.

C. References:

Sanders, K., "Documentation of Coating on Stainless Steel Plate" Memo to G. Gellatly, 19 June 1989.

PROJECT NUMBER: 1752
PROJECT TITLE: Molecular Structure Determination
PROJECT LEADER: G. Vilcins
WRITTEN BY: N. Jensen
PERIOD COVERED: June, 1989

I. MEASUREMENT OF INTERSTITIAL OXYGEN IN FILLER

- A. Objective: To determine the amount of interstitial oxygen in filler.
- B. Results: A series of real time experiments have been performed using a residual gas analyzer mass spectrometer which include studying the interaction of oxygen-18 and carbon dioxide atmospheres with filler and the gas evolution from filler at reduced pressure. Measurement of the changes in oxygen-18, oxygen-16, nitrogen and water with time serves to mark the transfer of gases between the filler and void volume of the container.
- C. Conclusions: To date, no data have been obtained which reveal an appreciable difference in accessibility between gases in the interstitial volume of the tobacco and the void volume of the container.
- D. Plans: Experiments are still in progress.
- E. References:
1. Jensen, N., PM Notebook #8620, p. 90.
 2. Shafer, K., PM Notebook #8789.

II. ANALYSIS OF CRS STEMS

- A. Objective: To analyze volatile components produced from CRS stems.
- B. Results: Samples of CRS stems and controls were analyzed by thermal chromatography - mass spectrometry. Pyrolysis was carried out in two steps and evolved components were chromatographed after each step on a DBWAX capillary column. The samples were heated to 150°C in step one and to 270°C in step two.
- C. Conclusions: An number of differences were observed between the chromatograms of the stems subjected to the ART process and the controls.
- D. Plans: The analysis of the data is in progress.

PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: June, 1989

ANALYTICAL FLAVOR SPECIFICATIONS

A. Objective: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities. To provide analytical certification on export flavors manufactured at the Flavor Center for FRG compliance.

B. Results:

The analysis of Direct Materials for compliance to FRG continues to require a large commitment of time. We are working in conjunction with Purchasing to certify on a priority basis those materials scheduled to be ordered. In addition, revised and preshipment samples are also received and analyzed following the samples for Purchasing to determine acceptability from a certification standpoint.

Specific lots of materials are being identified to formulate the PMI sample to reduce the potential for contamination of the final blend.

An investigation was conducted on some customer complaint samples from the Hong Kong market for compliance to manufacturing specifications.

A series of nicotine samples were analyzed by the HPLC procedure and the results were compared to those obtained by the Griffith distillation procedure. The distillates were also run. A memo was issued.

Samples of isosweet were analyzed by both the Flavor Center and the Flavor Specification Group to generate monitor-like samples to be used to check the methodology and instrument performance at the Cabarrus QA lab.

C. Plans: Continue the certification of the PMI samples, begin the certification of all flavors currently on hand at the Flavor Center and continue the specification work for the second vendor.

PROJECT NUMBER: 1759
PROJECT TITLE: Materials Evaluation & Elemental Analysis
PROJECT LEADER: P. F. Grantham
PERIOD COVERED: June, 1989

I. MATERIALS EVALUATION

A. **Objective:** To identify components of commercial products prior to their use at PM facilities.

B. **Results:**

Presentations on the Materials Evaluation program were made at York Engineering and the Make/Pack Equipment Standardization Committee meeting.

Tours were conducted at MC Primary, Park 500, York Engineering and Bermuda Hundred Processing Facility. Routine analysis of commercial products and Packaging Development samples continued.

D. **Plans:** Tours will be made at other facilities as scheduled. Presentations will be made as requested.

II. ELEMENTAL ANALYSIS

A. **Objective:** To provide qualitative and quantitative elemental data on tobacco, cigarette paper, development and materials evaluation samples.

B. **Results:**

X-ray fluorescence (XRF) was used to determine elemental concentrations of primarily, Mg, Ca, K, and Al in paper samples. Many of these samples were also analyzed using atomic absorption (AA). Routine tobacco samples, including silo storage study leaf samples were analyzed this month using both XRF and AA.

Paper samples submitted by S. Baldwin for project TRIM were analyzed to determine potassium concentrations using the potassium selective ion electrode (SIE) method.

Thermograms of multiple carbon samples using standard Thermogravimetric Analysis (TGA) conditions were generated to determine if a correlation between lightability and thermogram properties exist. The carbons were scanned between 25-700°C with a constant heating rate of 20°/min starting at 25°C. No definite correlation between lightability and TGA parameters was observed.

Evaluation of the XRF paper method is nearly complete. Final evaluation of some Mg data is currently underway.

D. **Plans:** Completion report for the XRF paper method will be written. Support to other R&D projects for elemental analysis will continue.

PROJECT NUMBER: 1902
PROJECT TITLE: Cell/Tissue Culture Research
PROJECT LEADER: I. L. Uydess
WRITTEN BY: J. B. Jones
PERIOD COVERED: June, 1989

I. ALTERNATE PRESERVATIVES PROGRAM - PHASE I SCREENS

- A. Objective: To develop procedures and to conduct microbiological screens to evaluate nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.
- B. Results: Previous Phase I shake-flask assays have been conducted with B. coagulans as the test organism. However, in order to broaden the scope of this assay, two additional Bacillus species commonly associated with tobacco (licheniformis and brevis) have recently been added to this assay to more thoroughly assess the efficacy of the compounds evaluated. Based upon this, a Phase I shake-flask screen of β -cyclocitrylidine acetic acid (β -CAA) was recently conducted using B. coagulans and B. licheniformis. Each organism was subjected to 50, and 150 μ g/ml β -CAA as well as the standard 150 μ g/ml propylparaben control. The results indicated that while both organisms were completely inhibited by the 150 μ g/ml dose of β -CAA, B. licheniformis was somewhat (10-20%) less sensitive than B. coagulans to both the 150 μ g/ml parabens control as well as the lowest dose of β -CAA that was tested.
- C. Plans: Additional tests will be conducted to evaluate the effectiveness of prospective preservative candidates against a variety of tobacco-identical bacteria.
- D. Reference:
Tenhet, S. W. Notebook No. 8281, p. 115.

II. ALTERNATE PRESERVATIVES PROGRAM - PHASE III SCREENS

- A. Objective: To evaluate changes in the organic acid composition of Park 500 SEL during lab-induced spoilage in 5-liter fermentors.
- B. Results: A modified HPLC procedure has been developed to better resolve citric, malic, succinic, lactic, formic, acetic, propionic, and butyric acids in fresh and lab-spoiled SELs. Lactic acid was observed to accumulate rapidly (4-fold over the baseline value) during the first 6-8 hours of spoilage. This was coincident with the drop in pH that is characteristic of this early stage of spoilage. The malic acid peak, on the other hand, decreased 2-fold during this same interval. A peak of unknown composition (retention time = 23.85 minutes) was also found to decrease substantially (≥ 10 -fold) during this time. However, as the pH of the spoiling SEL rose during the second phase of spoilage, acetic acid was found to accumulate progressively while the peak areas associated with many of the other volatile organic acids decreased

(presumably due to their utilization by the bacteria active during this period). These methods are being employed to better define the chemical changes that occur during spoilage as part of the overall efforts of the alternate preservative program.

- C. Plans: Continue additional investigations of this type and identify the chemical composition of the unknown peak at 23.85 minutes.

D. Reference:

Weissbecker, L. Notebook No. 8822, p. 35.

III. TOBACCO MICROBIOLOGY - ART STORAGE STUDY

- A. Objective: To develop and employ methods to evaluate the microflora resident in tobacco materials.
- B. Results: Four and six-week samples of the five cigarette models prepared for the ART Storage Study (see May Monthly) were removed from their storage facilities and evaluated for microbiological changes. Additional samples were submitted to CI and ARD Personnel for physical and chemical evaluation.
- C. Plans: The final evaluation of these materials is scheduled to occur on July 5, 1989.

D. References:

Chadick, D. Notebook No. 8825, pp. 1-15, 17-32.

Gaines, O. Notebook No. 8690, pp. 126-128, 130-132, 133, 135, 140-141, 143-146.

IV. SALMONELLA/MICROSOME (S/M) ASSAY: CROSSED SOLUBLE/BASE WEB STUDY - EFFECT OF NITROGEN

- A. Objective: Evaluate the effects of increased nitrogen via the addition of a model protein, amino acids and/or ammonium acetate to BuCEL/BrBW.
- B. Results: The percent added nitrogen was equivalent for each of the nitrogen sources used. There were some difficulties observed during the preparation of the CSCs; therefore not all of the prepared samples were smoked nor did all smoked samples receive four replicate smokings as is normally required in this assay.

Although there were some exceptional circumstances surrounding this study, a preliminary evaluation of the data revealed that: (1) there were no significant differences among any of the controls; (2) all of the test samples were significantly more active than the controls (particularly the samples treated with the amino acid mixture); (3) test samples prepared with ammonium

acetate were generally the lowest in S.A.; and (4) at comparable levels, the addition of protein and/or amino acids to the BrCEL + BrBW produced IT CSCs with significantly higher S.A.s than the addition to BrBW alone. There were no statistically significant differences observed using ammonium acetate with BrBW alone versus BrCEL + BrBW.

C. Plans: To expand this study by evaluating the effect(s) of addition of sugars/amino acids complex to BrCEL/BrBW.

D. Reference:

Thompson, L. H. Notebook No. 8731.

V. S/M ASSAY: CROSSED SOLUBLE/BASE WEB STUDY - BURLEY SOLUBLE (BUS1) FRACTIONS

A. Objective: To determine S/M IT CSC S.A. from two fractions of the BuS1 that have been subjected to reverse phase chromatography by M. Core and R. Izac.

B. Results: There were no differences among the control (BuS1) and the two test fractions.

C. Plans: There are no plans to further fractionate BuS1 using this methodology.

D. Reference:

Thompson, L. H. Notebook No. 8731.

PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Biochemistry
PROJECT LEADER: D. J. Ayers
WRITTEN BY: E. D. Mooz
PERIOD COVERED: June, 1989

I. LOW NICOTINE STUDY

- A. Objective: To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) step and specifically to isolate PMT from tobacco root extracts.
- B. Status: Tobacco plants from Group 18 were harvested and the roots and leaves (top, mid-stalk, and bottom) from 3, 5, 6, and 7 days after topping were stored at -80°C . Three roots (about 1 kg) from each of the 4 time periods were processed through the 40-65% ammonium sulfate stage and the extracts were assayed for PMT activity to find the optimal time for root harvest, which coincides with maximal PMT activity. These results will be compared with the data obtained from a similar study with Group 17 plants (1).

Four 100 ml aliquots of M-8-phenyl-Sepharose extract were applied to an SAH column for purification of PMT by affinity chromatography. The PMT active fractions were eluted with 1 mM SAM and stored at -80°C . for further use (2). Fifty ml of SAH-Sepharose was prepared for purifying additional PMT extracts (2,3).

Cytochrome c was chosen as an agent to minimize PMT-activity losses due to denaturation and/or nonspecific adsorptions in dilute PMT preparations. Amicon ultrafiltration and Centricon cells (not dry Sephadex-G-25) were found to be satisfactory methods for the concentration of PMT. A DEAE-5PW (TosoHaas) column with HPLC was observed to give an 100% yield of PMT activity. One mg of PMT preparation (M8- Φ -SAH-4) was concentrated and applied to a series of HPLC columns (anion exchange, hydroxyapatite, hydrophobic interaction, and gel permeation). The fractions containing PMT activity were pooled and concentrated after each column and before application to the next column. SDS-PAGE examination of fractions from the last column (gel permeation) indicated low protein levels of a nonhomogeneous preparation (4).

ELFE (preparative electrophoresis) investigations included the determination of the proper running conditions and loading efficiencies for different size gels. Preliminary experiments indicate that individual standard proteins may separate into specific fractions after elution from the gel. Studies are under way to check the feasibility of renaturing PMT after exposure to SDS (5).

Work has commenced on the isolation of total RNA from tobacco root and leaf tissue. Root tissue from tobacco plants grown hydroponically have been frozen and shipped to Stratagene for a cDNA library construction. A memo has been written on the use of differential hybridization for the isolation of root specific genes (6).

C. Plans: Process root and leaf material from Group 18 plants. Purify M-8- Φ extracts through SAH columns for use in other studies. Explore the four HPLC columns in series for the isolation of purified PMT. Confirm the sensitivity of the Protein Gold assay by the addition of standard protein to test solutions. Continue studies with ELFE and protein renaturation. Isolate, purify, and characterize RNA from tobacco roots.

D. References:

1. Lyle, J. Notebook No. 8397.
2. Mooz, E. D. Notebook No. 8803.
3. Yu, T. Notebook No. 8806.
4. Nakatani, H. Y. Notebook No. 8384.
5. Davies, S. M. Notebook No. 8761.
6. Malik, V. S. Notebook No. 8274.

PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: June 1989

I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES (Fournier, Paine, Podraza, Seeman)

- A. Objective: To develop inorganic materials for novel applications for reduced sidestream and enhanced subjectives in cigarettes and for required properties in novel smoking materials.
- B. Results and Plans: A series of CaX_2 ($\text{X} = \text{Cl}, \text{OAc}$) + M_2CO_3 ($\text{M} = \text{Na}, \text{K}$) reactions were performed, some in the presence of a dispersing agent (glycerol, diethylene glycol) to form CaCO_3 gel/particles. Gel stability was quantified by time for phase separation (immediate to 60 min). The particle sizes were determined by SEM and ranged from 0.01-6.00 microns with different morphologies.

Vanillin was found to be incorporated into the layers of hydrotalcite while eugenol, raspberry ketone, and isovanillin were not. Tube furnace pyrolysis of the hydrotalcite-vanillin product gave low yields of veratraldehyde, presumably due to an unprecedented transmethylation reaction within the layers of the inorganic moiety. A variety of magnesium, potassium and calcium double carbonates were prepared. A large scale preparation of calcium ethoxide was prepared per request from A. Kallianos.

The catalytic effects of iron oxides are being examined. Three samples with iron concentrations of 0%, 2% and 5% in alumina were prepared by codecomposition of the nitrates at 425°C. After further pulverization these samples will be submitted for papermaking as well as other standard characterization techniques. The alumina/iron oxide sample sent for x-ray diffraction indicated the sample to be amorphous. A sample has been submitted to Physical Research for confirmation. Microscopy of this same sample showed iron present in all crystallites (0.02 - 0.05 μm) examined. Another sample of $\text{AlOOH}/5\%\text{FeOOH}$ has been prepared by adding the appropriate amount of iron nitrate to Union Carbide's boehmite, peptizing, and precipitating the solids with KOH.

II. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Secor, Seeman)

- A. Objective: To develop techniques to remove selectively nicotine and other alkaloids from aqueous tobacco processing fluids.
- B. Results and Plans: We have continued to examine CR-2824, a tris(2-ethylhexyl)-derivative of tartaric acid as the liquid ion exchanger (LIX) for the membrane-based extraction process. This LIX results in excellent and reproducible nicotine extraction efficiencies. A large scale preparation of CR-2824 was completed.

Careful studies have shown that some of this LIX is transferred to the Tobacco Extract Solution (TES), even though it is nearly insoluble in water. We estimate that ca. 0.96 g of this LIX was transferred from the Organic Solvent (OS) to the TES during four 4-L runs (16 L of TES), or ca. 60 mg/L of TES. We conclude that it is the slight water solubility of the nicotine-LIX complex which drives the transfer, and that it may be possible to decrease the overall transfer to TES by back-transfer to OS by running each extraction to a greater percentage completion. This hypothesis will be tested shortly.

III. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Secor, Seeman)

- A. Objective: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.
- B. Results and Plans: A series of six benzylamines were prepared and will soon be sent to E. Bernstein. These are being investigated as analogs of nicotine and nornicotine. We are in the process of examining the clusters of these benzylamines with CO₂ and H₂O, to add experimental information regarding the relative extractability of the tobacco alkaloids in the ART process. Final reports are being written on benzyl alcohols, styrenes, and alkylpyrazines.

IV. FLAVOR/ODOR CHEMISTRY (Podraza)

- A. Objective: To prepare new substances for flavor/odor evaluation.
- B. Results and Plans: The natural product 3,6-dimethyl-3a,4,5,7a-tetrahydro-2(3H)-benzofuranone (CR-2818) was prepared in a two step process. A 0.5 gram sample of CR-2818 was delivered to Tom Gannon in Flavor Development for subjective evaluation. 6-n-Butylhexahydro-2(3H)-benzofuranone was prepared as a mixture of diastereomers (trans/cis ratio, 10/90).

PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. Comes
PERIOD COVERED: June, 1989

I. SIDESTREAM SMOKE

- A. **Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.
- B. **Status:** 1.) Evaluation of the six models in the analytical-subjective study continues by several methods. Multiple smoke runs have been carried out to independently collect, analyze and compare the SS gas phase and SS semivolatile fractions from these models. The semivolatile analysis by capillary gc-NPD/FID on the samples from silica gel fractionation is nearing completion. Additional runs using the Tekmar desorber for gas phase collection and analysis are in progress. 2.) The mixture of sulfur gases for calibration of the Sievers sulfur detector has been received. The setup of the detector is in progress and will continue as time permits. Development of procedures for analysis of sulfur compounds in MS and SS smoke will then be carried out. 3.) Testing of the first 8-port visibility apparatus continues in conjunction with personnel from U.S. Testing. The second 8-port system is nearing completion incorporating some design modifications to facilitate future planned operation in a QA environment. Assistance in this redesign/construction is currently being supplied on an as needed basis. A patent application is being written. 4.) Smoke distribution data has been obtained on two models (control and Mg(OH)₂ wrapped) from the analytical/subjective study using ¹⁴C-labelled sugars as the ¹⁴C-source. A 50/50 mixture of (U)-¹⁴C-glucose/fructose was spray applied to filler pulled from the appropriate models. This labelled filler was then remade into cigarettes using the same paper tubes. The smoke distribution study to compare the control with the small particle size CaCO₃ wrapped cigarette is about to begin.

II. SIDESTREAM SMOKING CHAMBER

- A. **Objective:** Design and construct an environmentally controllable chamber to measure selected components of sidestream smoke.
- B. **Status:** The major project effort during this reporting period has been on the chamber construction. Assistance has been required and generously supplied from several areas external the Chemical Research Division. Many modifications have been made to the HVAC equipment, to computer hardware and software necessary for chamber operation and data collection, to instrumentation for on-line data collection and to the laboratory itself. Preliminary smoking runs

have been made to study smoking machine operation and to investigate all parameters currently operational. Chamber smoke runs for "hard data" generation will commence shortly.

III. MISCELLANEOUS

Limited gc/ms and pyrolysis/gc/ms support was supplied as requested. Due to the priority of the Sidestream Smoke Chamber, a reassignment of personnel has been required. This has necessitated the temporary cessation of the gc/ms capability.

PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: Y. Houminer
PERIOD COVERED: June, 1989

I. COMMERCIALIZATION OF GMC AND POLYMIC

- A. **Objective:** To find chemical manufacturers who are capable of producing large quantities of GMC and polyMIC.
- B. **Results:** Analysis for trace amounts of solvents in the 1987 batch of glucose menthol carbonate (GMC) has been completed by Analytical Research. A decision has been made to change the solvent used in the PPG production process of menthyl chloroformate (MCR). In that connection a trip was made (by W. B. Edwards) to PPG Industries in LeForte, Texas, to discuss various aspects of their MCF production process.

The storage problem of MCF has been solved: Lee Labs agreed to store at their site any excess of menthyl chloroformate.

The search for a potential manufacturer of polyMIC continues. A meeting was held with a group from Eastman Kodak to discuss the status of their work on alternative methods for the synthesis of the monomer. While there seems to be no success in their work, they plan to continue research at the current pace.

A trip to Bayer AG is scheduled for late June to discuss with their R&D staff different technical aspects associated with polyMIC production.

II. FLAVOR RELEASE TECHNOLOGY

- A. **Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.
- B. **Results:** A significant progress has been made in the development of a direct synthesis of 4,6-O-acetal derivatives of free glucose. For example, the reaction of glucose with cinnamaldehyde dimethyl acetal in DMF using the resin IR-120 as catalyst gave good yield of the corresponding 4,6-O-acetal. This method is not only novel but also the only existing efficient method for synthesizing this class of compounds. The preparation of the corresponding acetals of α -amyl- and α -hexylcinnamaldehyde is now in progress.

We have carried out TGA (with the cooperation of Mr. C. Chung) on a series of acetal derivatives of glucose. It is concluded that distillation is the primary mode of weight loss for methyl 4,6-O-benzylidene- α -D-glucopyranoside (Aromatek 255) and methyl 4,6-O-(β -hexylstyrylmethylene)- α -D-glucopyranoside (Aromatek 245); a combination of pyrolysis and distillation for 4'-formyl-2'-methoxyphenyl 4,6-O-(styrylmethylene)- β -D-glucopyranoside (CR-

2831) and for 4'-formyl-2'-methoxyphenyl 4,6-O(β -amylstyrylmethylene)- β -D-glucopyranoside (CR-2833); and pyrolysis only for 4,6-O-(styrylmethylene)-D-glucose (CR-2834).

Curie Point pyrolysis GC/MS analysis (as performed by Mr. D. Magin) showed that at 315° and 595°C, there is only a 3 fold increase in the release of α -hexylcinnamaldehyde from Aromatek 245 at the higher temperature. On the other hand, under the same condition CR-2831 released 11 fold more cinnamaldehyde at the higher temperature. Furthermore, CR-2833 released the same amount of cinnamaldehyde at both temperature, as much as CR-2831 did at the higher temperature. These results support the conclusion that was reached from the TGA work.

III. FLAVOR CHEMISTRY

- A. **Objective:** To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.
- B. **Results:** The diethylamide and pyrrolidine amide of 2,2-dimethylvaleric acid have been prepared. These two similar amides have very different odor properties as was previously observed with the same amides of 2-ethylhexanoic acid. The N,N-diethyl-2,2-dimethylvaleramide has an intriguing combination of black pepper odor and menthol cooling.

The synthesis of the known black pepper compound is underway. Isobutyric acid was alkylated with 1-bromobutane to give 2,2-dimethylhexanoic acid. This has been converted to the corresponding acid chloride for later treatment with diethylamine.

Large numbers of cigarettes treated with various potential sidestream flavorants have been made and are presently being evaluated by the E-2 smoke panel.

IV. MISCELLANEOUS

The problem of ART cigarettes soiling has been investigated. Two sets of cigarettes, both of which contained spotted paper, were analyzed for tobacco waxes, primarily for solanesol. HPLC showed that the amount of solanesol was about two order of magnitude greater than that found on normal cigarette paper. This research is aimed at finding a method to reduce the soiling on paper found in the production of ART cigarettes. A sample of perhydrogenated solanesol was supplied to Analytical Research for use as an internal standard in the determination of solanesol.

Work continues on the separation of ART tobacco extracts in search for a fraction that contained a possible naturally occurring nicotine release agent.

One Kg of Oriental tobacco was extracted with hexane and dichloromethane. The extracts were submitted to S. Haut for further testing as possible nitrosamine inhibitor.

HPLC work, in support of other projects, has been carried out: the nicotine content of greenhouse grown Zinnia leaves was determined. Several attempts were made to find a HPLC separation method for the positional isomers, 2-ethyl-5-methyl and 2-ethyl-6-methylpyrazine.

PROJECT NUMBER: 2525
PROJECT TITLE: Paper and Tobacco Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: June, 1989

I. SIDESTREAM REDUCTION (S. Baldwin, S. Tafur, B. Rogers, G. Newell,
P. Suiter and G. Bokelman)

- A. Objectives: (1) Prepare and evaluate, for reduction in sidestream smoke, paper handsheets that contain coatings and/or inorganic fillers with different physical and chemical properties and (2) evaluate machine-made cigarettes containing cigarette papers produced at the University of Maine.
- B. Results: The fabrication of handmade cigarettes from handsheets prepared at PM and cigarette papers prepared at the University of Maine was continued. The types of cigarette papers and sizings included: (1) additional levels of potassium-containing fluxing agents on CaCO_3 and MgCO_3 papers, (2) soluble calcium in the form of calcium acetate, (3) hydrotalcite combined with CaCO_3 to improve ash appearance, and (4) Mg(OH)_2 bilayers and composites. For the month, 65 handmade cigarette models were prepared and sidestream analyses were obtained on 48 of these models.

Encouraging initial results for sidestream reduction, with apparently acceptable subjectives, have been obtained from the use of KH_2PO_4 as a sizing agent. Models have been evaluated in which the KH_2PO_4 was sized on the inner liner of dual-wrapped cigarettes, on high basis weight papers and on bilayer papers. In some cases reductions in sidestream comparable to those obtained with 35% Mg(OH)_2 papers have been achieved. Preliminary results have also indicated that KH_2PO_4 may have utility as an ash conditioner.

Similar handmade cigarettes with the Kimberly-Clark low sidestream paper (sized with 4.5% $\text{K}_2\text{succinate}$, 0.4% NaCMC and 0.5% MAP) were made with low CaCO_3 inner liners of both low and high porosity that had been sized with KH_2PO_4 . The dual-wrapped cigarette with the more porous inner liner (~55 Coresta) was preferred subjectively and gave a better reduction in sidestream visibility. These cigarette models provide a potential means for utilizing the Kimberly-Clark low sidestream paper with acceptable subjectives in a Lotus configuration.

S. Baldwin visited Dr. Allan Myerson in order to examine gas diffusion experimental capabilities which he has developed at New York Polytechnic University (1). These experimental techniques, if applicable to cigarette papers, would provide a direct measure of the diffusional properties of the papers of interest rather than an indirect one such as the electrolytic conductance technique used by Kimberly-Clark to estimate these properties. Paper samples were left with Dr. Myerson for preliminary evaluation of the feasibility of application of his techniques to cigarette paper.

Since a decision recently was made to switch back to Marlboro filler for handmade cigarettes, a comparison was conducted of

cigarettes containing 2I or Marlboro filler prepared from a series of experimental papers. Analyses indicated no differences in sidestream visibilities or burn times. Thus the change to Marlboro filler can confidently be made with no concern about comparisons to previous data generated on cigarettes with 2I filler.

BET surface area analyses were continued for filler samples submitted by A. Kallianos. These included: (1) ten alumina sol gel and Reheis alumina samples that had been submitted to heat treatments up to 1,000° C, (2) precipitated zirconia sol gel, (3) magnesium zirconate and (4) precipitated CaCO₃ sol gel.

Handmade cigarettes with papers incorporating "Smellrite" (a proprietary molecular sieve from Union Carbide) were evaluated by R. Southwick on the sidestream odor panel. No differences in sidestream aroma were detected between the "Smellrite" cigarettes and control cigarettes. Evaluation of the ability of "Smellrite" to effect cigarette butt odor is in progress.

Handmade cigarettes were prepared from paper handsheets containing 30% KMgPO₄. No significant reduction in sidestream visibility was found.

- C. Plans: More porous cigarette papers will be treated with KH₂PO₄ to see if significant reductions in sidestream visibility can be achieved. As soon as evaluation of these and existing cigarette models has been completed, we will consider how best to develop a proprietary patent position for the use of KH₂PO₄. Other acidic sizing chemicals and their levels of addition in single- and dual-wrapped cigarettes also will be examined. Studies involving the addition of various salts to tobacco filler will be continued. Finally, additional mono-layer and bilayer cigarette papers will be prepared at the University of Maine.

D. Reference:

1. Baldwin, S. D., memo to Dr. E. B. Sanders, "Gas Diffusion Experimental Capabilities at New York Polytechnic University," June 8, 1989.

II. GREENHOUSE STUDIES & SUPPORT ACTIVITIES (R. Bass, G. West & G. Newell)

- A. Objectives: Maintain the R&D greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs and provide requested assistance for special projects.
- B. Results: The hydroponic Burley 21 plants of Group 18 for Project 1904 were harvested. These plants produced a total root tissue yield of 18.47 kg with an average yield per plant of 362 g. The 52 hydroponic Burley 21 plants of Group 19 have been transplanted.

A group of 22 plant grafts was completed as part of a study on minor tobacco alkaloids.

The daily greenhouse plant production tasks have been done on schedule including the seeding and transplanting of Burley 21, Speight G-28, K-236 and Oriental.

In support of the Lotus program, a quantity of 2I filler was sprayed with a ^{14}C -labelled glucose and fructose sugar solution. Following equilibration, this filler was used to prepare 75 handmade cigarettes that were supplied to R. Newman for smoking.

- C. Plans: Maintain plant production schedules. Inspect and monitor plant and environmental conditions on a daily basis.

PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
WRITTEN BY: F. S. Hsu
PERIOD COVERED: June, 1989

I. PROJECT ART

- A. Objective: Evaluate the effects of the Mercedes and Muratti 2000 filters for the reduction of MS ammonia and other N-heterocyclics of ART cigarettes.

B. Results:

The filters from the ART cigarette (D9TC-1, 11 mg model) were removed and filters from Mercedes and Muratti 2000 cigarettes were reattached to the ART cigarette rods under the supervision of B. Hendricks. The filters of the ART cigarettes were removed and reattached to provide a control. The cigarettes were smoked after conditioning and with the ventilation holes taped. The results of the MS total and gaseous ammonia deliveries (5 determinations each) are given below.

Sample	Total Ammonia		Gaseous Ammonia		% Gaseous	
	<u>ug/cigt</u>	<u>% Reduced</u>	<u>ug/cigt</u>	<u>% Reduced</u>	<u>in Total</u>	
Monitor 25	26± 2	-	2.9 ±0.2	-	10	
Control	53± 8	-	15 ±2	-	30	
Mercedes	56±14	0	10 ±2	25	20	
Muratti	25± 2	55	9 ±1	40	35	

The ART cigarette with the Mercedes filter (dual plug with silica gel in the inner plug) showed a 25% reduction in gaseous ammonia, no reduction in total ammonia and the greatest reduction of N-heterocyclics in basic fraction of smoke compared to the control. The ART cigarette with the Muratti 2000 filter (plug-space-plug with carbon and silica gel between the plugs) showed a 40% reduction in gaseous ammonia, a 55% reduction in total ammonia and a selective reduction of some volatile bases and enhancement of minor alkaloids. The gaseous ammonia delivery of Muratti modified ART model was similar to that for the Mercedes modified ART model.

- C. Conclusions: The carbon in the Muratti 2000 filter appears to be more effective in removing protonated ammonia and N-heterocyclics. The silica gel in the Mercedes filter seems to have little effect on the total ammonia delivery and displays some selectivity toward basic smoke constituents.
- D. Plans: Provide filters with silica gel, silica gel/carbon, and carbon only (received from J. Piade, FTR) to Development (G. Keritsis) for further testing of ART models.
- E. References:

1. Parrish, M., PM Notebook 8729, pp. 40,44,58, & 62-63.
2. Hsu, F., PM Notebook 8786, pp. 66-69.

3. Parrish, M., Hsu, F., "MS Ammonia Delivery and TPM Basic fraction Analysis of ART Cigarettes with Mercedes and Muratti 2000 Filters," Memo to J. Charles, June 21, 1989.

- A. **Objective:** Determine the effect of treated ART models on the MS/SS gaseous ammonia delivery.
- B. **Results:** The MS/SS gaseous ammonia deliveries were measured for Art cigarettes treated with additives on the filler rod and the filter. The 8 mg ART cigarette models ("Next") had been modified as follows: 8802-16A, control sample treated with 20 μ L of water on the filler rod; 8802-16B, test sample treated with 20 μ L of 1N acetic acid (1.2 mg/cigt.) on the filler rod; 8802-16C, test sample treated with 30 μ L of 5% vanillin in ethanol on the filter (1.5 mg by filter weight); and 8802-16D, control sample treated with 30 μ L of ethanol on the filter.
- C. **Conclusions:** There were no significant differences in the MS or SS gaseous ammonia deliveries between the treated models and their respective controls. However, there may be a slight reduction in MS gaseous ammonia for the acetic acid treated sample (16B) compared to the control (16A).
- D. **Plans:** Confirm the difference of MS delivery between samples 16A and 16B.
- E. **References:**
1. Parrish, M., PM Notebook 8729, pp. 73-76, 78.
 2. Parrish, M., "MS/SS Gaseous Ammonia Delivery of Modified ART Cigarettes," Memo to G. Keritsis, June 20, 1989.
 3. White, G., "Total Ammonia Delivery in Mainstream Smoke of Control and Treated ART Cigarettes," Memo to G. Keritsis, June, 1989.

- A. **Objective:** Identify the source of the off-taste character of ART stems.
- B. **Results:** The following cigarettes, made with ART stems replacing all the Burley stems in RLTC, were analyzed to see if the smoke data can give any indication of the off-taste character of the ART stems.

<u>CIGARETTES</u>	<u>DESCRIPTION</u>
X6D9AOG	Control RLTC
X6D9AOH	Blended ART Runs
X6D9AOI	ART-Top Basket
X6D9AOJ	ART-Bottom Basket
X6D9AOK	Citrate Sprayed (not extracted)

Each cigarette was fractionated in triplicate into neutral, basic and acidic fractions and analyzed by gas chromatography. The mean peak areas, scaled to per mg condensate weight basis, were compared.

- C. Conclusions: The data from all three fractions showed the same change among the cigarette samples without any smoke constituents to be the apparent contributors.
- D. Plans: To locate the source of the off-taste by using smoke analysis, it is necessary to obtain information on how the "off-taste" from ART stems is defined. Studies will be conducted by smoking the test cigarettes with and without a Cambridge filter to assess the relative contribution of gas phase and semi-volatile components.
- E. References:

Hsu, F., Buckner, M., "Smoke Analysis of ART Stem Cigarettes",
Memo to B. Handy, June 22, 1989.

II. BURLEY SPRAY

- A. Objective: Determine the interaction mechanisms of Burley Spray with burley tobacco.
- B. Results:
- Two sampling methods capable of detecting a wide spectrum of organic compounds from BS were tested. The first one was a headspace technique involved purging the volatiles from 10 g of BS to a mixed sorbents of Tenax and Carbotrap and followed by thermodesorption and GC/MS analysis. The second method, for higher molecular weight compounds, used solvent extraction, concentration and Direct Chemical Ionization (DCI) analysis with ammonia or isobutane as reagent gases.
- The first stage experiment involved the heating of BS to boiling as a function of time to see any chemical reactions occurred. A two-stage condenser was used to prevent loss of volatiles and water content. A few differences detected by the headspace method are currently being identified by GC/MS.
- C. Conclusions: Headspace and DCI/MS analyses were tested to follow the composition changes of BS induced by temperature. The two methods can detect organic compounds of wide molecular weight range.
- D. Plans: Identify and rationalize the composition change of BS caused by temperature. Secondly, investigate the reaction products of BS with the introduction of ammonia, amino acids or organic acids with and without temperature.
- E. References:

Hsu, F., PM Notebook 8586, pp. 127-132.

PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: June, 1989

I. PROJECT ART

A. Objective: Provide analytical support to project ART.

B. Results:

The two aging studies using ART filler and ART cigarettes are still in progress. Results will be reported upon completion of the studies in July.

Determinations of nicotine in cigarette filler from cigarettes made at the MC were provided for Quality Engineering. The purpose of these tests was to determine if ART filler picked up nicotine from the makers. Results showed that no additional nicotine was incorporated into the ART cigarettes after one to two minutes of running the maker.

The amount of nicotine in liquid trap and bomb samples from the Bermuda Hundred pilot plant was determined.

C. Plans: Continue analysis of the samples from the two aging studies. Provide support to the ART processing facility as needed. Finish work on stem analysis and update method.

II. RESPONSE TO ANALYTICAL REQUESTS

A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by project personnel during the month of June included:

Non-routine packaging materials were quantitatively and qualitatively analyzed for residual solvents.

Several samples of burley tobacco were analyzed for methamidophos. No methamidophos was detected in any of the samples at a minimum detectable quantity of 0.5 ppm.

The amounts of benzoflex, dimoran, and triacetin in filter rods were determined for samples submitted by Gus Keritsis.

A recently developed HPLC procedure for the determination of methoprene on tobacco was used to quantitate the amount of methoprene picked up by tobacco processed in D pilot plant after application of Dianex® in the pilot plant. Detectable levels

(>0.5 ppm) were observed only for those samples which contacted areas intentionally sprayed. Some of the samples which contained detectable levels of methoprene were also analyzed for residual xylenes (part of Dianex® formulation). These samples contained less than 0.3 ppm xylenes.

The amount of Aromatek 245 was determined in cigarette paper and sidestream adhesive. This compound had intentionally been added as a potential SS odorant.

The amount of decanoic acid present in three RL samples submitted by Dan Teng was determined. The samples contained between 400 and 545 ppm.

Several customer complaint samples were analyzed this past month. One sample contained moderately high levels of tax stamp ink solvent.

PROJECT NUMBER: 6902
PROJECT TITLE: Biochemical Special Investigations
PROJECT LEADER: B. D. Davies
PERIOD COVERED: June, 1989

I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

- A. **Objective:** To obtain a monoclonal antibody (MCA) against nicotine (NIC-MCA).
- B. **Results:** Preliminary screening of spent media from 14 clones was completed using standard assay procedures. Several were shown to react specifically with nicotine coupled to thyroglobulin. None of these interactions was inhibitable by nicotine (1×10^{-4} M) under the standard conditions. A modification to the standard procedure, involving increased nicotine concentrations and dilutions of the spent media (antibody), has been introduced and has increased the sensitivity of the assay. Preliminary assessment of data from assays using the modified protocol do show moderate levels of nicotine inhibition of binding.
- C. **Plans:** Further examine the sensitivity of the modified screening assay and quantitate the nicotine inhibition of binding of spent culture media from clones. Implement modified assay to fully screen clones.
- D. **References:**
Crockett, E. Notebook No. 8783, p. 100.

II. ADDITIONAL APPROACHES TOWARD PUTRESCINE METHYLTRANSFERASE (PMT) ISOLATION

- A. **Objective:** Provide additional experimental approaches to assist in the effort to isolate PMT.
- B. **Results:** An ELFE fractionation was performed on a mixture of PMT enzyme active material and photoaffinity labeled ^3H -PMT. The bulk of the PMT enzyme activity was recovered in fractions 7 and 8, mimicking previous results from our Project and Project 1902. Protein bound ^3H was also recovered in fractions 7 and 8. This indicated that under native electrophoresis separation procedures ^3H -PMT and native PMT migrate coincidentally.

An experiment was performed to examine the total level of incorporation of SAM in PMT/PAL experiments utilizing a high specific activity/low concentration versus a low specific activity/high concentration ^3H -SAM solutions. The results of that experiment indicated that more SAM was incorporated into PMT using low specific activity solutions. However, as expected, lower levels of ^3H were incorporated.

- C. Plans: Repeat the ELFE experiment and determine if larger quantities of ^3H -PMT can be obtained. Prepare larger quantities of the low specific activity, ^3H -SAM, PAL/PMT material and determine if less ^3H is lost during subsequent fractionation procedures.

D. References:

Crockett, E. Notebook No. 8783, p 100.
Dunn, R. Notebook No. 8721, pp. 120-140.

PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: J. M. Penn
WRITTEN BY: G. J. Patskan
PERIOD COVERED: June, 1989

I. PROTEIN KINASE C (PKC) ASSAY IN INTACT CELLS

- A. Objective: To examine the response of quiescent 3T3 cells to 12-O-tetradecanoylphorbol-13-acetate (TPA), 2R1 CSC and X6D5RN CSC in the PKC intact cell assay.
- B. Results: The data from an experiment using 19.5 hr treatments of TPA at 1 and 10 ng/ml and CSC at 20 and 100 ng/ml were processed and analyzed. TPA had no effect at 1 ng/ml while 5- to 6-fold increases in protein phosphorylation were observed at the 10 ng/ml concentration. At 20 ug/ml 2R1 CSC did not effect phosphorylation, however X6D5RN CSC caused a 1.5- to 2.0-fold increase. The 100 ug/ml concentration of 2R1 CSC elicited a 1.5-fold increase in protein phosphorylation and X6D5RN produced a 3.5-fold increase. In each case where increased phosphorylation was detected no one protein was phosphorylated to a greater extent than another.
- C. Conclusions: The rank order of activity in the protein phosphorylation assay does not correlate with other bioassay data.
- D. Plans: This assay will be discontinued and a completion report will be written.
- E. Reference:
- Nixon, G. M. Notebook No. 8711, p. 123.

II. ACQUISITION AND MAINTENANCE OF CELL LINES

- A. Objective: To acquire and maintain a variety of cell lines for use in biochemical assays.
- B. Results: The cultures of 3PC, MT1/2 and V79 cells are growing well. A large batch of MT1/2 cells were preserved in liquid nitrogen. High laboratory temperatures resulted in the loss of the cell cultures of the 3T3 cells and the three clones of JB-6 cells. New cultures of these cells have been started from frozen stocks.
- C. Plans: Monitor and characterize the new cultures of 3T3 and JB-6 cells.
- D. References:
- Burruss, T. J. Notebook No. 8804, p. 127.
Stagg, D. L. Notebook No. 8553, p. 168.
Vaughan, B. G. Notebook No. 8828, p. 196.

III. EGF BINDING ASSAY

- A. Objective: To examine the role of phospholipid metabolism in the inhibition of EGF binding caused by CSC.
- B. Results: One experiment was completed which examined the effects of phospholipase C, a treatment which should liberate diacylglycerol, on EGF binding at 30 minutes and 19.5 hr. Phospholipase C had no significant effects on EGF binding, whereas the positive controls reduced binding. An experiment was also conducted to examine the effects of indomethacin on CSC's capacity to inhibit EGF binding. Indomethacin, an inhibitor of cyclo-oxygenase activity, did not counteract the effect of 2R1 CSC. However, considerable variation in the cell count data was noted in this experiment.
- C. Plans: Repeat these experiments after the new culture of 3T3 cells has been established.
- D. Reference:
- Stagg, D. L. Notebook No. 8553, p. 168.

IV. PDBU BINDING ASSAY

- A. Objective: To determine the effects of TPA and 2R1 CSC on the binding of ³H-PDBu to 3T3 cells.
- B. Results: A third experiment was completed which examined the effects of a 2 hr pretreatment with TPA or 2R1 CSC at 4°C. The results of this experiment were similar to the previous ones. TPA inhibited binding while CSC had only minimal effects. This binding protocol does reduce the effect of 2R1 CSC on nonspecific binding which had been observed in typical time of binding treatment experiments. In another experiment the 2R1 CSC was given for 19.5 hr at 37°C prior to the measurement of ³H-PDBu binding, a typical pretreatment experiment. As in previous experiments the effects of 2R1 CSC were minor under these conditions.
- C. Conclusions: 2R1 CSC does not significantly affect the binding of ³H-PDBu to 3T3 cells.
- D. Plans: Compare the effects of 2R1 CSC and X6D5RN CSC on ³H-PDBu binding after both 4°C and 37°C incubations once the new culture of 3T3 cells has been established.
- E. Reference:
- Burruss, T. J. Notebook No. 8804, p. 127.

V. GLUTATHIONE DEPLETION ASSAY

- A. Objective:** To evaluate the role of arachidonic acid metabolism in the reduction of glutathione by 2R1 CSC in V79 cells.
- B. Results:** Incubation of the cells with dibromoacetophenone (25 - 200 ug/ml), an alkylating agent which inhibits phospholipase A₂ activity, augmented the effects of 2R1 CSC (0.17 - 1.0 mg/ml). Treatment with indomethacin (1 - 100 uM), an inhibitor of cyclo-oxygenase activity, enhanced the effects of 2R1 CSC but only at the lowest dose of indomethacin.
- C. Plans:** Reexamine the effects of indomethacin and test the effects of nordihydroguaiararetic acid, a lipoxygenase inhibitor, on the response to 2R1 CSC.
- D. Reference:**

McCoy, W. R. Notebook No. 8739, p. 99.

PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: June, 1989

I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Data collection was continued as part of an aging experiment on water-washed burley (Bu) filler. The study is nearly complete at this time. After 126 days at 45°C there has been no change in alkaloids, but a ten-fold increase has occurred in endogenous NNK. The samples stored at -30°C were unchanged. MS TSNA determinations have not been completed.

Microencapsulated nicotine bitartrate (μ NBT) was added to low alkaloid burley tobacco (LA Bu) as a model for unextracted nicotine. This experiment was designed to determine whether the release of nicotine at temperatures above those at which it would be distilled normally would result in the formation of NNK during smoking. LA Bu (J5BUV) was chosen as a substrate for the experiment because of its high nitrate and low endogenous alkaloid levels, which result in low MS TSNA deliveries with the potential for TSNA pyrosynthesis. MS TSNA levels from the LA Bu with 280°C releasable μ NBT were increased by ~40% over control LA Bu. Corresponding increases of ~50-60% occurred when 405°C releasable μ NBT was evaluated. However, replication of this experiment is needed to verify these results.

- C. Plans: The final sample for the washed burley aging study will be analyzed in July, and this study will then be complete. The μ NBT experiment will be repeated.
- D. Reference:

Haut, S. A. Notebook No. 8768, p. 151.

II. UNEXTRACTED NICOTINE STUDIES

- A. Objective: To determine whether there is a correlation between unextracted nicotine (UN) in base web (BW) and the delivery of NNK in the corresponding MS smoke.
- B. Results: The initial objective is to measure the levels of total alkali-releasable nicotine (TARN), extractable nicotine (EN), water-soluble UN (Nic-X), and water-insoluble UN (Nic-Y) in three BWs which have a known delivery of MS NNK. The three BWs are BuBW, bright (Br) BW and oriental (Or) BW from the Crossed Solubles Base Web Study. Analysis for nicotine in the extracts is being carried out by HPLC using a cyano column and a methanol-dimethylethanolamine phosphate buffer. TARN is determined by extraction of the BW with 1N NaOH at 90°C for 24 h, and analysis

of the extract. An extraction with water at 90°C for 24 h removes EN and Nic-X. The marc from the latter extraction contains Nic-Y. Nicotine is measured in this aqueous extract, followed by hydrolysis at 90°C for 24 h, whereupon nicotine is again measured. The difference between the nicotine levels before and after hydrolysis represents Nic-X. Methanol (MeOH) extraction at 22°C for 24 h removes EN, leaving Nic-Y (and possibly Nic-X) in the marc. Preliminary results from this study have shown that BrBW contains more TARN, EN (water), and EN (MeOH) than either BuBW or OrBW. BuBW contains the most Nic-Y, followed by BrBW and OrBW. Extraction with alkali for times longer than 24 h continue to produce more nicotine, and the time will have to be extended to ensure complete removal.

- C. **Plans:** More experiments are planned and are now being carried out in order to obtain additional data with the objective of achieving a nicotine balance. Alkali extracted BWs have been submitted to ARD for estimation of nicotine remaining by pyrolysis-MS. Isolation of Nic-X from CEL will be attempted. The water-insoluble marcs will be treated with various enzymes to render Nic-Y soluble enough for isolation. Attempts will be made to identify Nic-X and Nic-Y. Plans will be made to correlate Nic-X and Nic-Y with NNK formation.

D. **References:**

Lambert, E. A. Notebook No. 8523.
Keene, C. K. Notebook No. 8754.

III. **ORIENTAL INHIBITOR STUDIES (ORGANIC EXTRACTS)**

- A. **Objective:** To determine whether the causative agent(s) responsible for the reduced levels of MS TSNA observed for Or tobacco can be removed with organic solvents and applied to other fillers as a means of decreasing the MS TSNA levels delivered by these fillers.
- B. **Results:** Four extracts of blended oriental tobacco were prepared by R. Izac to be evaluated for their effect on MS TSNA. The extractions were carried out in a Waring Blender. One batch of tobacco was extracted first with methylene chloride (MeCl) followed by MeOH. A second batch of tobacco was extracted with hexane (HEX) followed by MeCl. The extracts were applied to two experimental fillers: (1) ExBu (Bu tobacco extracted with 5% ethanol/HEX) is depleted in nicotine and endogenous TSNA, but contains minor alkaloids and nitrate and is, therefore, a good substrate for pyrosynthesis; (2) BuBW is low in endogenous TSNA, nicotine, minor alkaloids and nitrate, but produces pyrosynthetic NNK from an unknown precursor. BuBW is a good substrate for evaluating the effect of additives on NNK formation. The results obtained on smoking cigarettes made with the treated ExBu fillers were not conclusive. There were some reductions in MS TSNA relative to the corresponding control, but they were not very large and not reproducible. On a ng/cigarette basis both the HEX and

MeCl initial extracts gave small reductions which also held true when the data were converted to ng/mg TPM for the most part. None of the secondary extracts gave consistent reductions, but tended to give increases in MS TSNA, due to the presence of considerably higher levels of minor alkaloids found in these extracts than in the initial extracts. When the initial MeCl extract was reevaluated the reductions could not be observed. The HEX extract was not reevaluated. On BuBW, the initial MeCl extract gave a reduction in MS TSNA on a ng/cigarette basis, but this reduction did not hold true when the data were calculated on a ng/mg TPM basis. The initial HEX extract was not evaluated on BuBW.

- C. Plans: Several samples of Or filler will be extracted by Supercritical Fluid Processing, Inc. in July using supercritical CO₂ under varying conditions to produce extracts that will be tested for inhibition of TSNA pyrosynthesis. Later these extracts will be fractionated in attempts to isolate and identify an inhibitor.

D. Reference:

Haut, S. A. Notebook 8768, p. 151.

IV. TSNA IN PROJECT ART MATERIALS

- A. Objective: To determine the TSNA levels of fillers with lowered nicotine content or other material generated in Project ART studies.
- B. Results: Three supercritical CO₂ extracted DL5 blend fillers were submitted, along with a control, for MS and filler TSNA analysis. The extractions were carried out by Project 1704 in the 1 L extractor using bright stems, carbon, and a water column as CO₂ scrubbers, respectively, for the three extractions. The filler analyses showed that the use of stems as a scrubber resulted in ~52-62% removal of TSNA, while water and carbon as scrubbers resulted in ~92-97% removal of TSNA. On a ng/cigt. basis, use of the stem scrubber gave a reduction in MS NNN and NAT of 30 and 40%, respectively, while NNK was reduced by 13%. On the same basis, NNN and NNK were reduced by ~55%, while NAT was reduced by ~67% using either water or carbon scrubbers. On a ng/mg TPM basis, stems provided reductions of 11 and 22% for NNN and NAT, respectively, while NNK increased by 14%, which was taken as indicating no change. Water gave a lower TPM value than obtained using carbon as a scrubber, and this caused a differentiation of the results when calculated on a ng/mg TPM basis. Therefore, reductions of 57, 68, and 58%, respectively, were obtained for NNN, NAT, and NNK using carbon as a scrubber, while corresponding reductions of 34, 51 and 33% were obtained using water as a scrubber. A memo discussing this experiment is in preparation.

C. Reference:

Tickle, M. H. Notebook No. 8716.

V. MISCELLANEOUS AND SUPPORT STUDIES

- A. Objective: To conduct studies of the TSNA content of filler and/or MS smoke or carry out other activities as necessary to support other PM programs.
- B. Results: A method with improved safety precautions was devised for spraying various solutions onto specific fillers supplied by another group. The sprayings were completed. Tests were conducted on collection methods and analysis for TSNA and VNA in the sidestream chamber. Several problems were encountered, but calibration of the chamber is now being carried out.

C. References:

Haut, S. A. Notebook No. 8768, p. 151.
Morgan, W. R. Notebook No. 8579, p. 187.

PROJECT NUMBER: 6912
PROJECT TITLE: Tobacco/Smoke Relationships
PROJECT LEADER: S. B. Hassam
WRITTEN BY: S. Drew
PERIOD COVERED: June, 1989

I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Thin layer radiochromatography (TLRC) of MS TPM extracts from radiolabeled nicotine cigarettes was continued. As described in the April monthly summary, the MS TPM buffer solutions were extracted with methylene chloride. Preparative chromatography of methylene chloride extracts was done on silica gel plates (1000 μ thickness) in methylene chloride/methanol (20/1). The LSC of bands obtained from preparative TLC of smoke samples showed that for smoking #1: of the radioactivity applied to the plates, 24% was associated with a broad region (zone 2) including an area co-chromatographed with NNK, and 53% with a more polar zone. Of the activity applied to the plate, ~77% was recovered. In smoking #2: of the radioactivity applied to the plates, ~80% was recovered. Of the recovered activity, 18% was associated with zone 2 (as described above), and 61% with a more polar zone.

Based on integration of the scans, it is estimated that 0.01 - 0.05% of the initial activity present in the cigarettes (50 μ Ci) may be associated with a peak that co-chromatographs with NNK. RP-HPLC in conjunction with radioactivity detection confirmed that zone 2 is a mixture of 14 C compounds, and that there is activity associated with retention times that corresponds to NNN/NNK (12.0 - 16.0 min.). (Reference mixtures of labelled TSNA and nicotine showed good resolution).

GC/TEA analysis of zone 2 from radiolabeled smoke samples showed NAT, NNN and NNK present. However, compared to the amounts determined for nonradiolabeled cigarettes, the loss of 70% of the expected TSNA delivery was indicated for the 14 C cigarettes. Some of this may be attributed to difficulties in smoking, collecting, and isolation methods.

- C. Plans: Analyze filler blend used for making cigarettes for nicotine and NNK content. Determine if study needs to be repeated, and refine smoke collection methods as needed.
- D. Reference:

1. Hassam, S. Notebook No. 8823, pp.

II. CROSSED SOLUBLES/BASE WEB STUDY (CHEMISTRY)

- A. Objective: To investigate the smoke chemistry of model cigarettes made from all possible combinations of solubles from bright, burley and oriental tobaccos on base webs from the three tobaccos.

- B. Results: Bright CEL with added nitrogen (in the form of amino acids, a protein or ammonium acetate) was sprayed on bright base web and made into cigarettes. In addition to the smokings of nitrogen addition samples reported last month, control sample smokings for the study were completed. The CSC from each smoking was submitted for testing in the S/M assay.

A sample of burley CEL insolubles was sprayed on to bright base web. The RL (Bu11/BrBW) was made into cigarettes, and attempts to smoke these cigarettes were successful.

Puff counts determined on calcium acetate addition cigarettes show that the puff count increases significantly from 10±1 to 13 when the amount of calcium added is ≥6% (total solids held constant).

Samples of burley CEL treated previously with various ion exchange resins were extracted with different solvents. Eight samples were produced. Based on sample dry weight the bulk of the CEL solubles is recovered in the neutrals. The dry sample from each extraction was dissolved in water. Of the eight samples produced six were sprayed on bright base web and made into model cigarettes.

- C. Plans: Preparation of fillers and cigarettes will be continued. Cigarettes will be smoked for the Salmonella/microsome assay. Formulation of plans for C Pilot Plant production of RL will be completed. Preliminary experiments to demineralize CEL using the electrodialysis unit will be continued.

D. References:

1. Hellams, R. Notebook No. 8613, pp. 163-164
2. Drew, S. Notebook No. 8899, pp. 29-37.

III. SUPPORT FUNCTION: CONDENSATE PREPARATION

- A. Objective: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.
- B. Results: Handmade cigarettes were prepared for Project 6908, 1902, 1620 and 6912. Cigarettes were smoked on a 30-port Borgwaldt smoking machine to collect CSC for S/M and EGF assay testing.
- C. Reference:

1. McGee, N. Notebook No. 8743, pp. 47-63.

PROJECT NUMBER: 0008
PROJECT TITLE: CAD - Computer Technology
PROJECT LEADER: R. Lipps, M. Allred, N. Latif
PERIOD COVERED: June, 1989

I. Chemical Analysis Lab System

- A. **Objective:** Develop a lab support environment for the Chemical Analysis Section including instrument support, sample receiving support, data reporting, and accounting.
- B. **Results:** Version 1 of the Chemical Analysis Laboratory Support project was completed in May. This version's official release was accompanied by the creation of a release copy of the software, the deinstallation of the HP/1000 LIMS system and a final meeting with the Chemical Analysis Support team. This system, conceptually patterned after the current CTSD laboratory support system and providing a complete set of workstation/host cooperative systems, delivers enhanced capabilities including instrument run independence from a host environment, a standard style of supporting laboratory data and a consistent tool set for data control, editing, reduction and reporting. This system has made reported data available to high level analysis tools like RS/1 through electronic data transfer and direct interaction with a database of finished data.

This product provides the Chemical Analysis section with increased performance options, reliability and storage capacity while eliminating the obsolete HP 1000 system. The system is fully supported by the Computer Applications Division allowing for ease of modification and enhancement. The database design allows for flexibility in data retrieval and presentation. Its modular design accomodates the ability to add additional instrumentation with minimal software effort. Also, many of the concepts and development work done here are directly applicable to the modernization of the CTSD laboratory support system.

The current release includes the following features:

- Inhouse utilities provide Sample Receiving request logging, test specification, and label support under the Freeform database/screen tool and the ShareBase database platform.
- Worklists are generated on the host, downloaded to the instrument workstation and used to schedule testing. Results are reported locally. Approved results are uploaded to the hosts and incorporated into the ShareBase database.
- Design features of the system allow for instrument workstations to be run independently from a host computer. [New]

- PC instrument workstations interact with technicians and with instruments to collect test results. IBM PC/XT microcomputers and in-house Microsoft Quick Basic utilities are used for gravimetric analyses. IBM PC/AT-compatibles and PE Nelson chromatography systems are used for the chromatographic and the continuous flow analyses. Results from these instrument workstations are concentrated on a central host using PC/NFS over ethernet. [New]
 - Data storage is provided in an environment conducive to editing, screening, reporting and approving.
 - Final data reports are automatically generated at the conclusion of the entry of data values. [New]
 - Data Reporting and distribution is provided via electronic mail as requested. [New]
 - Finished data is distributed to a long-term storage database. RPL procedures allow authorized personnel to extract this ShareBase data into RS/1. [New]
 - An electronic accounting system has been established for tracking sample/test volume in the Chemical Analysis section. [New]
- C. Plans: Requested enhancements for the current system will be handled via the project request system and incorporated on a prioritized and scheduled basis in a formal release cycle.

PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Jane Y. Lewis
PERIOD COVERED: June, 1989

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. Japan - New Brands

F. J. Burrus, Inc. introduced Parisienne Mild King Size (10 mg tar, 0.7 mg nicotine) and Pierre Cardin Lights 100 (4 mg tar, 0.4 mg nicotine) on April 1.

The House of Craven introduced Craven "A" Super Mild 100 Box (9 mg tar, 0.6 mg nicotine) and Craven "A" 100 Box (12 mg tar, 1 mg nicotine) on April 1.

2. Japan - 17 mm Circumference Cigarettes

Japan Tobacco, Inc. introduced Epique Superslim 100 Menthol cigarettes on April 1. This cigarette delivers 9 mg tar, 0.9 mg nicotine and 0.4 mg smoke menthol.

American Cigarette Company, Ltd. introduced Vogue Superslims 100 (plain and menthol) cigarettes. These cigarettes deliver 8 mg tar, 0.7 mg nicotine and 0.3 mg smoke menthol.

II. INDUSTRY MONITOR

A. Objective: To produce a monitor cigarette (four million units) to be used as an industry-wide monitor for smoking analyses (TPM, nicotine and water).

B. Results: The monitor was manufactured in Semiworks June 7-13. The cigarettes were randomized and canned the week of June 26.

During production of the monitor, a random sample from each of the 17 individual pallets was obtained. These 17 individual samples were tested and the overall average of the results are as follows:

Tar, mg/cigt.	17.5
Nicotine, mg/cigt.	1.15
Weight Control, %	
±20 mg	91
±25 mg	98
Firmness, mm	2.78
Oven Volatiles, % (As Is)	13.63

- C. Plans: The monitor will be calibrated internally and then distributed industry-wide for collaborative calibration.

III. DYNAMIC RTD/VENTILATION

- A. Objective: To obtain the capability of generating RTD and ventilation data on cigarettes during smoking.
- B. Results: Operating parameters for the RTD and ventilation while smoking instrument have been optimized. Capillary standards, certified by the Standards Laboratory, yielded significantly lower values on the dynamic smoker. This can be explained by the way the puff is taken on the smoker. The sine function predicts a peak flow of 1600 cc/min. on the smoker, whereas, 1050 cc/min. constant flow is generated on the static instruments.

The length of the capillary standards used is not long enough to establish laminar flow and due to the mechanics of the smoking machine, the machine will never behave as the Pressure Drop instruments used for total RTD measurements. The results obtained from the capillaries are reproducible from day-to-day and can be used to monitor the instrument's operation.

Preliminary puff-by-puff for the RTD and ventilation of monitor cigarettes, M#25 and LDM#5, have been obtained.

- C. Plans: Continue testing of monitor cigarettes to establish trends in puff-by-puff profiles and to assure consistent operation of the dynamic instrument.

IV. NICOTINE IN SMOKE FOR ART CIGARETTES

- A. Objective: Develop methodology to analyze samples with a low nicotine to tar ratio and analyze samples as needed.
- B. Results: A method and a completion report have been written detailing the modifications to the GC procedure needed for measuring nicotine in smoke for ART cigarettes. Wide bore capillary chromatography is needed to clearly resolve nicotine in these samples that have a low nicotine to tar ratio. Precision was found to be 13% (relative standard deviation at 2-sigma) at nicotine levels of 0.04 and 0.07 mg/cigt. This indicates that nicotine is accurately reported to 3 places behind the decimal (2 significant figures) at these levels.

Nicotine results from the wide bore method were compared to those from the conventional GC methods used by CTSD and TITL. Results generally agree within 0.01 mg/cigt. with the wide bore results being slightly higher than either conventional method.

- C. Plans: This method is being certified by U.S. Testing. ART samples are being analyzed as needed using the new methodology.